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Set	Items	Description
S1	1780008	IC OR ICS OR ((INTEGRATED OR LOGIC) (W) (CIRCUIT? ?)) OR (MICRO) (W) (ELETRONIC? OR CIRCUIT? ? OR CHIP? ?) OR CHIP? ? OR MICROCIRCUIT? ? OR DIE? ? OR LOGIC(W) CIRCUIT? ? OR WAFER? ? OR MICROELECTRONIC OR DICE OR CC=B2220 OR CC=B2570
S2	83675	(COPPER OR CU) (2N) (METAL? OR STUD? ? OR PAD? ?)
S3	75900	(COPPER OR CU) (2N) (LAYER? OR FILM? OR COAT?)
S4	264402	ELECTRICAL? (W) CONDUCT?
S5	845	INTERMEDIATE (W) BARRIER OR (UNDER) (W) (BUMP OR PAD) OR UNDERPAD? OR UNDERBUMP?
S6	156297	POLYMERIC
S7	45595	POLYIMIDE
S8	1587	BENZOCYCLOBUTENE
S9	361	POLYBENZOXAZOLE
S10	844659	TIN OR PB OR SN OR INDIUM OR (TIN OR SN) (W) (LEAD OR PB OR - INDIUM OR SILVER OR AG OR BISMUTH OR BI)
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S12	324709	ISOPROPYL (W) ALCOHOL OR METHANOL OR GLYCOL OR METHY (W) PYRROLIDONE
S13	11	METHYPYRROLIDONE
S14	69145	(SI OR SILICON) (W) (NITRIDE OR N)
S15	6940	(SILICON OR SI) (W) (OXYNITRIDE OR MONONITRIDE) OR SION OR SILICON (W) NITRIDE (W) OXIDE
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S17	3179	AU=(SMITH, P OR SMITH P OR SMITH, PATRICIA OR SMITH PATRICIA)
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S23	53	S22 NOT S20
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S27	0	S25 AND DRY (W) CHEMICAL (W) VAPOR?
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S29	48	S28 NOT (S20 OR S23)

21/3,AB/1 (Item 1 from file: 2)
 DIALOG(R)File 2:INSPEC
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7394881 INSPEC Abstract Number: B2002-11-0170J-029
 Title: Electromigration failures of UBM/bump systems of flip-chip packages
 Author(s): Wu, J.D.; Zheng, P.J.; Lee, K.; Chiu, C.T.; Lee, J.J.
 Author Affiliation: Adv. Semicond. Eng. Inc., Kaohsiung, Taiwan
 Conference Title: 52nd Electronic Components and Technology Conference 2002. (Cat. No.02CH37345) p.452-7
 Publisher: IEEE, Piscataway, NJ, USA
 Publication Date: 2002 Country of Publication: USA xxxiv+1789 pp.
 ISBN: 0 7803 7430 4 Material Identity Number: XX-2002-01381
 U.S. Copyright Clearance Center Code: 0-7803-7430-4/02/\$17.00
 Conference Title: Proceedings of 52nd Electronic Components and Technology Conference
 Conference Sponsor: IEEE Components, Packaging, & Manuf. Technol. Soc.; Electronic Components, Assemblies & Mater. Assoc
 Conference Date: 28-31 May 2002 Conference Location: San Diego, CA, USA

Language: English

Abstract: A flip-chip package is studied in a high temperature operation life test to evaluate the structural integrity of **under bump** metallurgy (UBM) and solder bumps. To understand the impact of current polarity on solder bumps, a reverse engineering methodology is employed to calculate bump resistance histories having different current directions and metal trace resistance between two adjacent bumps. It is concluded that the observed high resistance increase is from bumps with electrical current flowing upward into the UBM/bump interface (cathode), while bumps having opposite current polarity cause only a minor resistance change. The direction of electron flow affects the reaction rates of UBM and flip-chip solder, resulting in different failure modes and degradation rates. The effects of current density and operation temperature are also probed. It is observed that operation temperature has a more direct and significant influence than current density on bump failures owing to the low melting point characteristics of eutectic solder. Failed test vehicles are subjected to cross section analysis via SEM, the identified failure sites are from aforementioned high resistance bumps with structural damage at the region of UBM and UBM/bump interfaces.

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7352020 INSPEC Abstract Number: B2002-09-0170J-097
 Title: Growth prediction of tin/copper intermetallics formed between 63/37 Sn/Pb and OSP **coated copper** solder **pads** for a flip chip application
 Author(s): Grilletto, C.; Arroyave, C.A.; Govind, A.; Salvaleon, E.R.
 Author Affiliation: LSI Logic Corp., Milpitas, CA, USA
 Journal: IEEE Transactions on Electronics Packaging Manufacturing vol.25, no.2 p.78-83
 Publisher: IEEE,
 Publication Date: April 2002 Country of Publication: USA
 CODEN: ITEPFL ISSN: 1521-334X
 SICI: 1521-334X(200204)25:2L:78:GPCI;1-6

Material Identity Number: H313-2002-003

U.S. Copyright Clearance Center Code: 1521-334X/02/\$10.00

Language: English

Abstract: This study quantifies the effect of temperature and time on the growth of Cu-Sn intermetallics, specifically for flip **chip**/ball grid array packaging technology. The activation energy and the growth rates were determined for solid state diffusion, after the initial assembly reflow(s). Three different types of solder joints were investigated. 1) BGA 63/37 solder joints which were formed by a standard convection oven attach of 30 mil (760 μ m) diameter solder spheres to OSP protected, Cu plated ball **pads** of an organic flip **chip** substrate. The ball pads are solder mask defined and of 0.635 mm nominal diameter. 2) Flip **chip** bump pad solder joint consisting of 63/37 eutectic solder bumped **die** attached to a nonsolder mask defined, OSP protected, Cu plated **pad** of the flip **chip** substrate. The flip **chip** bumps on the **die** are created by screen printing solder paste on the **die** pads and subsequent reflow attach, by a standard convection oven to the **die** under bump metallurgy (UBM). The nominal **die** UBM pad diameter is 0.085 mm. 3) Solder joint formed on a coupon which involved the reflow of the balls randomly placed on a Cu plated **layer** with no solder mask coating. The investigation was performed by first establishing the intermetallic growth rate at six different temperatures, ranging from 85 degrees C to 150 degrees C. The relationship between intermetallic growth and time was shown to essentially follow the common parabolic diffusion relationship to temperature especially above 100 degrees C. The activation energy (E/a) and the growth constant ($k/0$) were then calculated from this data. The results showed that the E for the total intermetallic thickness was essentially similar for the three solder joint configurations of the ball, bump and the coupon described above. E varied from 0.31 eV to 0.32 eV, while the $k/0$ varied from 18.0 μ m/s/ $\sup{1/2}$ to 24.2 μ m/s/ $\sup{1/2}$.

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DIALOG(R)File 2:INSPEC

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7157210 INSPEC Abstract Number: B2002-02-0170J-061

Title: Effect of Cu **stud** microstructure and electroplating process on intermetallic compounds growth and reliability of flip-**chip** solder bump

Author(s): Guo-Wei Xiao; Chan, P.C.H.; Teng, A.; Jian Cai; Yuen, M.M.F.

Author Affiliation: Dept. of Electr. & Electron. Eng., Hong Kong Univ. of Sci. & Technol., Kowloon, China

Journal: IEEE Transactions on Components and Packaging Technologies
vol.24, no.4 p.682-90

Publisher: IEEE,

Publication Date: Dec. 2001 **Country of Publication:** USA

CODEN: ITCPFB **ISSN:** 1521-3331

SICI: 1521-3331(200112)24:4L:682:ESME;1-Y

Material Identity Number: H324-2002-002

U.S. Copyright Clearance Center Code: 1521-3331/01/\$10.00

Language: English

Abstract: In electroplating-based flip-**chip** technology, the Cu **stud** and solder deposition processes are two of the most important factors affecting the reliability of solder joints. The growth of Cu-Sn intermetallic compounds (IMC) also plays a critical role. In this paper, the effect of Cu **stud** surface roughness and microstructures on the reliability of solder joint was studied. The surface

roughness of the **Cu stud** was increased as the Cu electroplating current density increased. The microstructural morphology of the **Cu-Sn IMC layer** was affected by **Cu stud** surface structure. We found the growth rate of IMC layer increased with the increasing of **Cu stud** grain size and surface roughness during aging test. The growth kinetics of Cu-Sn intermetallic compound formation for 63Sn/37Pb solder followed the Arrhenius equation with activation energy varied from 0.78 eV to 1.14 eV. The ratios of Cu/sub 3/Sn layer thickness to the total **Cu-Sn IMC layer** thickness was in the range of 0.5 to 0.15 for various Cu microstructures at 150 degrees C during thermal aging test. The shear strength of solder bump was measured after thermal aging and temperature/humidity tests. The relationship between electroplating process and reliability of solder joints was established. The failure mode of solder joints was also analyzed.

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7055529 INSPEC Abstract Number: B2001-11-0170J-185
 Title: **Cr/Cu/Ni underbump metallization** study
 Author(s): Tay Hui Leng; Galen Kirkpatrick; Andrew Tay; Lu Li
 Author Affiliation: Inst. of Microelectron., Singapore
 Conference Title: 2001 Proceedings. 51st Electronic Components and Technology Conference (Cat. No.01CH37220) p.939-44
 Publisher: IEEE, Piscataway, NJ, USA
 Publication Date: 2001 Country of Publication: USA xxxiii+1518 pp.
 ISBN: 0 7803 7038 4 Material Identity Number: XX-2001-01138
 U.S. Copyright Clearance Center Code: 0 7803 7038 4/2001/\$10.00
 Conference Title: 51st Electronic Components and Technology Conference 2001. Proceedings
 Conference Sponsor: Components, Packaging, & Manuf. Technol. (CPMT) Soc. IEEE; Electron. Components Assemblies & Mater. Assoc. (ECA); Electron. Components Sector of the Electron. Ind. Alliance
 Conference Date: 29 May-1 June 2001 Conference Location: Orlando, FL, USA

Language: English

Abstract: In flip **chip** interconnection using eutectic Pb/Sn solder bumps, a highly reliable **underbump** metallization (UBM) is required to maintain adhesion and solder wettability. An experimental study investigated the thermal stability of the Cr/Cu/Ni UBM-where Cr act as an adhesive, Cu a solder wettable layer and Ni a barrier. The process window for good thermal stability will reduce silicon cratering failure and intermetallic failure to ensure reliability. The **Cu** and **Ni layers** were varied in low, medium and high thickness to study their impact on solder bump strength and failure mechanisms. 5*3 mm full array test **chips** (with Cr/Cu/Ni UBM) were subjected to thermal stability tests (1) multiple reflow for 1x, 5x, 10x, 20x and (2) high temperature storage at 150 degrees C up to 1000 hrs. Destructive ball shear test and cross-sectional analysis was done. Bump shear results show that the Cr/Cu/Ni UBM, with Ni thickness (low to high) remains stable with respect to the number of reflow cycles. The failures were cohesive (Mode I-within solder). A high Ni thickness inhibited Cu diffusion and suppressed Cu IMC formation at near solder interface. Under high temperature storage, intermetallic growth was accelerated and the excessive intermetallic formed was very brittle. For low Ni thickness, failure mode (Mode I+ Mode II) was observed after aging (>500 hrs). Failure mode remained as cohesive in high Ni thickness UBM. For low to high thickness Cu mini-bumps, shear strength

was maintained during multiple reflows and the shearing fracture remains within the solder. Failure mode shifted from Mode I (at $t=0$) to Mode III interfacial failure (after aging) in Cr/low thickness Cu/medium thickness Ni UBM, when the limited Cu supply led to solder dewetting.

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7051905 INSPEC Abstract Number: B2001-11-0170J-068

Title: Development of an etchant for selectively etching TiWN/sub x/ in the presence of electroplated 95%Pb-5%Sn solder

Author(s): Ramanathan, L.N.; Mitchell, D.

Author Affiliation: Final Manuf. Technol. Center, Motorola Inc., Chandler, AZ, USA

Journal: IEEE Transactions on Components and Packaging Technologies vol.24, no.3 p.425-30

Publisher: IEEE,

Publication Date: Sept. 2001 Country of Publication: USA

CODEN: ITCFPB ISSN: 1521-3331

SICI: 1521-3331(200109)24:3L.425:DESE;1-H

Material Identity Number: H324-2001-003

U.S. Copyright Clearance Center Code: 1521-3331/2001/\$10.00

Language: English

Abstract: Shrinking **die** sizes and increasing I/O density is motivating the push toward flip **chip** packages. A flip **chip** interconnection system with a **under bump** metallurgy stack containing sputtered TiWN/sub X//sputtered **Cu**/electroplated **Cu stud**/electroplated 95%Pb-5%Sn was developed. An important step in the above process is the selective etching of the sputtered **Cu bus layer** and the TiWN/sub X/ barrier layer, in the presence of the Pb-Sn solder. The **Cu bus layer** was selectively etched using commercial etchants. However, no commercial etchants were available for selectively etching the TiWN/sub X/ layer, $H/sub 2/O/sub 2/-NH/sub 4/OH$ based etching systems, popularly known as Standard Clean-1 cleaning solutions, have been extensively used to clean silicon **wafers** in front end **wafer** fabrication where only trace metal contamination exists. Since **metals** like lead, **copper**, titanium, tin and tungsten catalyze the heterogeneous decomposition of the peroxide, the unstable $H/sub 2/O/sub 2/-NH/sub 4/OH$ based etching systems are rarely used to etch metal films. In this paper the development of a $H/sub 2/O/sub 2/-NH/sub 4/OH$ based etchant to selectively etch the sputtered TiWN/sub X/ films in the presence of electroplated 95%Pb-5%Sn solder bumps is discussed. A $2/sup 3/$ full factorial experiment with mid point was conducted to establish the etchant composition, as well as process temperature, that give satisfactory responses with respect to etch time, permissible undercut of the **Cu stud** (caused by the $NH/sub 4/OH$), and acceptable bump shape after reflow. Statistical analysis was used to understand the significant factors influencing the etch rate and undercut. An etchant containing 6% by volume of 30%- $H/sub 2/O/sub 2/$ and 0.75% by volume of 30%- $NH/sub 4/OH$ operated at a temperature of 37 degrees C was found to give satisfactory results.

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21/3,AB/6 (Item 6 from file: 2)

DIALOG(R)File 2:INSPEC

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6880887 INSPEC Abstract Number: B2001-05-2240-007

Title: Study on failure mode of solder bump fabricated using eutectic solder electroplating process

Author(s): Guo-wei Xiao; Chan, P.C.H.; Teng, A.; Jian Cai; Yuen, M.M.F.

Author Affiliation: Dept. of Electr. & Electron. Eng., Hong Kong Univ. of Sci. & Technol., China

Conference Title: International Symposium on Electronic Materials and Packaging (EMAP2000) (Cat. No.00EX458) p.18-26

Editor(s): Kim, J.K.; Teng, A.; Lee, S-W R.

Publisher: IEEE, Piscataway, NJ, USA

Publication Date: 2000 Country of Publication: USA xii+478 pp.

ISBN: 0 7803 6654 9 Material Identity Number: XX-2001-00192

U.S. Copyright Clearance Center Code: 0 7803 6654 9/2000/\$10.00

Conference Title: International Symposium on Electronic Materials and Packaging (EMAP2000)

Conference Sponsor: Adv. Interconnect Technol.; ASM Pacific Technol.; ASME Int. - Hong Kong Sect.; Compass Technol. Co.; US Army Res. Office - Far East; Inst. Mater. Res. & Eng. (IMRE), Singapore; Nanyang Technol. Univ. Singapore

Conference Date: 30 Nov.-2 Dec. 2000 Conference Location: Hong Kong, China

Language: English

Abstract: The electroplating-based flip **chip** process has many advantages over other solder bumping methods, where the bump fabrication process can affect the reliability of solder joints. In this paper, the effect of the UBM and electroplating process of the solder bump on the shear strength of the solder bump is studied, as well as the relationship between the shear test failure mode and solder bump fabrication process. It is reported that the Cu surface roughness is affected by the Cu plating current density and the appropriate current density is in a range from 10~40 mA/cm². The solder bump plating process temperature should be within 30-35 degrees C. It is observed that the growth kinetics of intermetallic compound formation are affected by the **Cu stud**

structure. The ratio of Cu/sub 3/Sn to the total **Cu-Sn IMC layer** thickness was from 0.5 to 0.15 with various Cu microstructures at 150 degrees C during thermal aging tests. The activation energy was in the range of 0.78 eV to 1.14 eV. Five shear test failure modes are analyzed which are related to the electroplating process.

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21/3,AB/7 (Item 7 from file: 2)

DIALOG(R)File 2:INSPEC

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6703480 INSPEC Abstract Number: B2000-10-0170J-070

Title: The effect of **Cu stud** structure and eutectic solder electroplating on intermetallic growth and reliability of flip-**chip** solder bump

Author(s): Guowei Xiao; Chan, P.; Cai Jian; Teng, A.; Yuen, W.

Author Affiliation: Dept. of Electr. & Electron. Eng., Hong Kong Univ. of Sci. & Technol., China

Conference Title: 2000 Proceedings. 50th Electronic Components and Technology Conference (Cat. No.00CH37070) p.54-9

Publisher: IEEE, Piscataway, NJ, USA

Publication Date: 2000 Country of Publication: USA xxxv+1756 pp.

ISBN: 0 7803 5908 9 Material Identity Number: XX-2000-01366

U.S. Copyright Clearance Center Code: 0 7803 5908 9/2000/\$10.00

Conference Title: 2000 Proceedings. 50th Electronic Components and Technology Conference

Conference Sponsor: Components, Packaging, and Manuf. Technol. Soc. of IEEE; Electronic Ind. Alliance

Conference Date: 21-24 May 2000 Conference Location: Las Vegas, NV, USA

Language: English

Abstract: In electroplating-based flip-chip technology, the Cu stud and solder deposition process is one of the most important factors affecting the reliability of solder joints. The growth of Cu-Sn intermetallic compounds (IMC) also plays a critical role. In this paper, the effect of Cu stud surface roughness and microstructures on the reliability of solder joint was studied. The micro-structural morphology of the Cu-Sn IMC layer was affected by Cu stud surface structure. The Cu stud with sloped edge can impact the adhesion of solder bump and UBM (Under Bump Metallurgy) layer. Insufficient solder wetting at edge of the Cu stud can further degrade the reliability of solder joints. We observed the thickness of epsilon -phase Cu/sub 3/Sn layer increased continuously instead of eta -phase Cu/sub 6/Sn/sub 5/ due to the deficiency of tin at the bottom of solder bump after extended thermal aging time increased. The adhesion of Cu stud and UBM layer was weakened due to the growth of Cu/sub 3/Sn at the edge of Cu stud. Both of the Cu-Sn IMC layers grew at the top of Cu stud as the aging time increased. The mean thickness of two IMC layers increased linearly with the square root of aging time. We found that cracks formed easily at the interface of Cu-Sn IMC layer and solder bump, especially at the Pb-rich layer and IMC layer interface. Cracks led to low bump shear strength after extended thermal aging. We did not observe cracks formed at the Cu/sub 6/Sn/sub 5/ and Cu/sub 3/Sn interface. The SEM and EDAX analysis suggested that the fracture surface structure was influenced by the Cu stud microstructure and solder bump deposition process.

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21/3,AB/8 (Item 8 from file: 2)

DIALOG(R)File 2:INSPEC

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6206153 INSPEC Abstract Number: A1999-09-8140N-076, B1999-05-2550E-048

Title: Direct correlation between mechanical failure and metallurgical reaction in flip chip solder joints

Author(s): Liu, C.Y.; Chih Chen; Mal, A.K.; Tu, K.N.

Author Affiliation: Dept. of Mater. Sci. & Eng., California Univ., Los Angeles, CA, USA

Journal: Journal of Applied Physics vol.85, no.7 p.3882-6

Publisher: AIP,

Publication Date: 1 April 1999 Country of Publication: USA

CODEN: JAPIAU ISSN: 0021-8979

SICI: 0021-8979(19990401)85:7L:3882:DCBM;1-9

Material Identity Number: J004-1999-005

U.S. Copyright Clearance Center Code: 0021-8979/99/85(7)/3882(5)/\$15.00

Language: English

Abstract: We tested flip chip solder bonded Si samples under tensile and shear loading as a function of annealing time at 200 degrees C. The solder bump was eutectic SnPb and the underbump thin film metallization was Cu/Cr deposited on oxidized Si. We found that the failure mode is interfacial fracture and the fracture strength decreases rapidly with annealing time. From scanning electric microscope

observations, the fracture occurs at the Cu-Sn/Cr interface. We conclude that it is the metallurgical reaction that has brought the solder into direct contact with the Cr surface. The weak joint is due to the spalling of Cu-Sn compound grains from the Cr surface, especially near the edges and corners of the joint.

Subfile: A B

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21/3,AB/9 (Item 9 from file: 2)

DIALOG(R)File 2:INSPEC

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6155268 INSPEC Abstract Number: B1999-03-0170J-036

Title: Maskless, direct deposition of copper onto aluminum bond pads for flip **chip** applications

Author(s): Fang, M.; O'Keefe, T.; Stroder, M.; Shih, W.; O'Keefe, M.; Strawser, R.; Via, D.

Author Affiliation: Missouri Univ., Rolla, MO, USA

Conference Title: Electronic Packaging Materials Science X. Symposium
p.85-90

Editor(s): Belton, D.J.; Gaynes, M.; Jacobs, E.G.; Pearson, R.; Wu, T.

Publisher: Mater. Res. Soc, Warrendale, PA, USA

Publication Date: 1998 Country of Publication: USA ix+262 pp.

ISBN: 1 55899 421 1 Material Identity Number: XX-1998-03239

Conference Title: Electronic Packaging Materials Science X. Symposium

Conference Date: 14-16 April 1998 Conference Location: San Francisco, CA, USA

Language: English

Abstract: Flip **chip** interconnection of **integrated circuits** (IC) for packaging applications such as direct **chip** attachment use Pb-Sn solders as the connection between the **die** and the substrate. **Underbump** metallization is typically used as a transition from the nonsolderable Al bond pad on the IC to a solderable surface such as copper using traditional blanket metal deposition, photolithography and etching procedures. In this study, we report for the first time the use of a novel process for selective deposition of adherent copper directly on to aluminum thin films, eliminating the need for adhesion promoting transition layers and additional patterning steps. Using copper bearing organic solutions and standard electroless and electrolytic copper plating baths, as-deposited and annealed sputter deposited Al-x%Cu (x=0 to 2) thin films were coated with **metallic copper**. An increase in the organically deposited copper nucleation site density was observed with increasing copper concentration in the sputtered aluminum/**copper** thin films. Preliminary results using focused ion beam microscopy indicated that dissolution of the aluminum oxide surface and subsequent deposition of copper by cementation occurs in the nonconducting organic solution at sub-micron reaction lengths. Qualitative adhesion testing of samples resulted in the majority of films passing the tape test. Demonstration of the process using 50 μ m diameter vias in BCB coated flip **chip** test vehicles from MCNC are presented.

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21/3,AB/10 (Item 1 from file: 8)

DIALOG(R)File 8:Ei Compendex(R)

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06289021

E.I. No: EIP03067351358

Title: Pb-free Sn/3.5Ag electroplating bumping process and **under bump** metallization (UBM)

Author: Jang, Se-Young; Wolf, Juergen; Ehrmann, Oswin; Gloor, Heinz; Reichl, Herbert; Paik, Kyung-Wook

Corporate Source: Samsung Electronics Company, Ltd., Suwon, South Korea

Source: IEEE Transactions on Electronics Packaging Manufacturing v 25 n 3 July 2002. p 193-202

Publication Year: 2002

CODEN: ITEPFL ISSN: 1521-334X

Language: English

Abstract: Pb-free solder is one of the biggest issues in today's electronic packaging industry. This paper introduces a newly developed Sn/3.5Ag alloy plating process for **wafer** level bumping. The effects of **Under Bump** Metallization (UBM) on the process, interfacial reaction, and mechanical strength have been investigated. Four different types of sputtering-based UBM **layers** - TiW/Cu/electroplated Cu, Cr/CrCu/Cu, NiV/Cu, and TiW/NiV - were fabricated with eutectic Pb/63Sn and Sn/3.5Ag solder. The result shows that the Sn/Ag solder gains Cu or Ni from UBM's and becomes Sn/Ag/Cu or Sn/Ag/Ni during reflow process. Sn/Ag solder has higher reactivity with Cu and Ni than Pb/63Sn. The Intermetallic Compound (IMC) spalling from the interface between UBM/solder has been observed on Cr/CrCu/Cu and TiW/NiV UBM's. However, the IMC spalling phenomena did not decrease the bump shear strength with a bump size of 110 μm , whereas a size of 60 μm brought a decrease in shear value and failure mode change. 27 Refs.

21/3,AB/11 (Item 2 from file: 8)

DIALOG(R)File 8: Ei Compendex(R)

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06261538

E.I. No: EIP03027308213

Title: Phenomena of electroless Ni-P and intermetallic-compound stripping and dissolving in Sn-Bi and Sn-Pb solder joints with Au/EN/Cu **metallization**

Author: Huang, Chien-Sheng; Yeh, Jih-Hung; Young, Bi-Lian; Duh, Jenq-Gong

Corporate Source: Dept. of Materials Science and Eng. National Tsing Hua University, Hsinchu, Taiwan

Source: Journal of Electronic Materials v 31 n 11 November 2002. p 1230-1237

Publication Year: 2002

CODEN: JECMA5 ISSN: 0361-5235

Language: English

Abstract: Electroless Ni-P (EN) has been popularly adopted and used as a diffusion barrier in the **under-bump** metallurgy (UBM) for flip-**chip** application. The EN with different P contents was first deposited on activated Cu/Al//20//3 substrates. To prevent the EN from oxidation, a thin Au coating was further plated on the EN/Cu/Al//20//3 substrates. Two types of solder alloys (63Sn-37Pb and 42Sn-58Bi) and two compositions of EN (Ni-5.5wt.%P and Ni-12.1wt.%P) were employed to investigate the interfacial reaction in the joint of solder/Au/EN/Cu. Occurrence of EN and intermetallic compound (IMC) stripping and dissolving was revealed. After annealing, Ni//3Sn//4 and Ni//3P formed between the solder and the EN in all joints. However, some of the Ni//3Sn//4 IMCs stripped into the solder for a longer annealing time. The stripped EN was first observed in the Sn-Bi/Au/Ni-5.5wt.%P/Cu/Al//20//3 joints annealed at 185 degree C for 180 min. The stripped IMCs and the EN then dissolved in the solder and formed the Ni-P-Cu-Sn-Pb solid solution in the Sn-Pb/Au/Ni-5.5wt.%P/Cu/Al//20//3 joints annealed at 200 degree C or 240

degree C. The phenomenon of IMC stripping was found in all joints. However, both the stripping and dissolving of EN was only observed in joints with Ni-5.5wt.%P. The tendency of IMC stripping was related to the amount of IMCs, while the EN stripping corresponded to the surface condition of the EN. 21 Refs.

21/3,AB/12 (Item 3 from file: 8)
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06202757

E.I. No: EIP02467212346

Title: Lead-free solder bump technologies for flip-**chip** electronic packaging applications

Author: Karim, Zaheed S.; Chow, Alice; Cheung, Edwin; Cheung, Gary

Corporate Source: Adv. Interconnect Technology Ltd., Tsuen Wan, Hong Kong

Conference Title: Physics of Semiconductor Devices

Conference Location: Delhi, India Conference Date: 20011211-20011215

E.I. Conference No.: 60210

Source: Proceedings of SPIE - The International Society for Optical Engineering v 4746 I 2002. p 570-575

Publication Year: 2002

CODEN: PSISDG ISSN: 0277-786X

Language: English

Abstract: Five different types of lead-free solder bumps consisting of pure-tin, tin-bismuth, tin-copper, tin-silver, and tin-silver-copper were fabricated. The method of fabrication for all lead-free alloys was by electroplating using a sputtered and **copper layer** followed by a thicker plated **copper under-bump-metal**. Results indicate that the application of a suitable barrier diffusion layer is necessary to limit the excessive formation of tin-copper intermetallic compounds. (Edited abstract) 4 Refs.

21/3,AB/13 (Item 4 from file: 8)
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05994269

E.I. No: EIP01446711676

Title: Cr/Cu/Ni **underbump metallization** study

Author: Leng, T.H.; Kirkpatrick, G.; Tay, A.; Li, L.

Corporate Source: Institute of Microelectronics, Singapore 117685, Singapore

Conference Title: 51st Electronic Components and Technology Conference

Conference Location: Orlando, FL, United States Conference Date: 20010529-20010601

E.I. Conference No.: 58652

Source: Proceedings - Electronic Components and Technology Conference 2001. p 939-944 (IEEE cat n 01CH37220)

Publication Year: 2001

CODEN: PECCA7 ISSN: 0569-5503

Language: English

Abstract: In flip **chip** interconnection using eutectic Pb/Sn solder bumps, a highly reliable **underbump** metallization (UBM) is required to maintain adhesion and solder wettability. An experimental study investigated the thermal stability of the Cr/Cu/Ni UBM - where Cr act as an adhesive, Cu a solder wettable layer and Ni a barrier. The process window for good thermal stability will reduce silicon cratering failure and intermetallic failure to ensure reliability. The Cu and Ni

layers were varied in low, medium and high thickness to study their impact on solder bump strength and failure mechanisms. 5 multiplied by 3 mm full array test chips (with Cr/Cu/Ni UBM) were subjected to thermal stability tests (1) multiple reflow for 1x, 5x, 10x, 20x and (2) high temperature storage at 150 degree C up to 1000 hrs. Destructive ball shear test and cross-sectional analysis was done. Bump shear results show that the Cr/Cu/Ni UBM, with Ni thickness (low to high) remains stable with respect to the number of reflow cycles. The failures were cohesive (Mode I-within solder). A high Ni thickness inhibited Cu diffusion and suppressed Cu IMC formation at near solder interface. Under high temperature storage, intermetallic growth was accelerated and the excessive intermetallic formed was very brittle. For low Ni thickness, failure mode (Mode I+ Mode II) was observed after aging (greater than 500 hrs). Failure mode remained as cohesive in high Ni thickness UBM. For low to high thickness Cu mini-bumps, shear strength was maintained during multiple reflows and the shearing fracture remains within the solder. Failure mode shifted from Mode I (at t=0) to Mode III interfacial failure (after aging) in Cr/low thickness Cu/medium thickness Ni UBM, when the limited Cu supply led to solder dewetting. 9 Refs.

21/3,AB/14 (Item 5 from file: 8)
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05785904

E.I. No: EIP01025527443

Title: Effect of **Cu stud** structure and eutectic solder electroplating on intermetallic growth and reliability of flip-chip solder bump

Author: Xiao, Guowei; Chan, Philip; Jian, Cai; Teng, Annette; Yuen, Matthew

Corporate Source: Hong Kong Univ of Science and Technology, Hong Kong

Conference Title: 50th Electronic Components and Technology Conference

Conference Location: Las Vegas, NV, USA

Source: Proceedings - Electronic Components and Technology Conference 2000. IEEE, Piscataway, NJ, USA, 00CB37070. p 54-59

Publication Year: 2000

CODEN: PECCA7 ISSN: 0569-5503

Language: English

Abstract: In electroplating-based flip-chip technology, the **Cu stud** and solder deposition process is one of the most important factors affecting the reliability of solder joints. The growth of Cu-Sn intermetallic compounds (IMC) also plays a critical role. In this paper, the effect of **Cu stud** surface roughness and microstructures on the reliability of solder joint was studied. The micro-structural morphology of the Cu-Sn IMC **layer** was affected by **Cu stud** surface structure. The **Cu stud** with sloped edge can impacted the adhesion of solder bump and UBM (**Under Bump Metallurgy**) layer. Insufficient solder wetting at edge of the **Cu stud** can further degrade the reliability of solder joints. We observed the thickness of epsilon -phase Cu//3Sn **layer** increased continuously instead of eta -phase Cu//6Sn//5 due to the deficiency of tin at the bottom of solder bump after extended thermal aging. The adhesion of **Cu stud** and UBM layer was weakened due to the growth of Cu//3Sn at the edge of **Cu stud**. Both of the Cu-Sn IMC **layers** grew at the top of **Cu stud** as the aging time increased. The mean thickness of two IMC layers increased linearly with the square root of aging time. We found that cracks formed easily at the interface of Cu-Sn IMC **layer** and solder bump, especially at the Pb-rich layer and IMC layer interface. Cracks led to low

bump shear strength after extended thermal aging. We did not observe cracks formed at the Cu//6Sn//5 and Cu//3Sn interface. The SEM and EDAX analysis suggested that the fracture surface structure was influenced by the Cu stud microstructure and solder bump deposition process.
(Author abstract) 12 Refs.

21/3,AB/15 (Item 6 from file: 8)
DIALOG(R)File 8: Ei Compendex(R)
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05759733

E.I. No: EIP01015487294

Title: Development of **under bump** metallizations for flip **chip** bonding to organic substrates

Author: Korhonen, T.M.; Su, P.; Hong, S.J.; Korhonen, M.A.; Li, C.-Y.

Corporate Source: Cornell Univ, Ithaca, NY, USA

Source: Journal of Electronic Materials v 28 n 11 Nov 1999. p 1146-1149

Publication Year: 1999

CODEN: JECMA5 ISSN: 0361-5235

Language: English

Abstract: Several **under bump** metallization (UBM) schemes using CuNi alloys as the solderable layer were investigated. Nickel slows down dissolution of the UBM into the solder and formation of intermetallics during reflow. To study the intermetallic reaction, CuNi foils of different concentrations were immersed in a eutectic PbSn solder bath for reaction times ranging from 30 seconds to 30 minutes. It was observed that when 10% and 20% Ni is added into copper, the intermetallic forms a continuous layer, instead of the discrete scallops seen in pure Cu/solder interfaces. However, the thickness of the intermetallic remained about the same. For 30% and 45% Ni alloys a definite decrease in the intermetallic thickness was observed compared to the lower Ni alloys. Actual **under bump** metallizations were also made on Si **wafers** to study the reactions when there is a limited supply of CuNi available. Cr or Ti was used as the adhesion layer, and the solderable **layer** was a **copper-nickel** alloy, instead of pure copper used in the conventional UBM scheme. The metal layers were deposited on a **wafer** by evaporation and patterned into contact pads. Eutectic PbSn solder balls were reflowed on top of the pads. SEM micrographs of the intermetallic that forms at the UBM/solder interface show the refining effect of Ni in the interfacial microstructure. Since nickel metallizations often have high stresses, stress in the UBMs was measured by the **wafer** curvature method. Stress vs Ni content plots show that while stresses increase somewhat with the Ni content, the adhesion layer under the CuNi layer has a much larger effect on the stress. UBMs with Cr/CrCu adhesion layer had stresses ranging from about 300 to 600 MPa, while the stresses in UBMs with Ti/TiNi layers were between 70 and 350 MPa. (Author abstract) 9 Refs.

21/3,AB/16 (Item 7 from file: 8)
DIALOG(R)File 8: Ei Compendex(R)
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05151329

E.I. No: EIP98114433255

Title: Flip **chip** metallurgies for lead-free solders

Author: Korhonen, T.M.; Hong, S.J.; Korhonen, M.A.; Li, C.-Y.

Corporate Source: Cornell Univ, Ithaca, NY, USA

Conference Title: Proceedings of the 1998 MRS Spring Symposium

Conference Location: San Francisco, CA, USA Conference Date: 19980414-19980416

E.I. Conference No.: 48894

Source: Electronic Packaging Materials Science X Materials Research Society Symposium - Proceedings v 515 1998. MRS, Warrendale, PA, USA. p 79-83

Publication Year: 1998

CODEN: MRSPDH ISSN: 0272-9172

Language: English

Abstract: The most commonly used lead-free solders contain large amounts of tin, which makes them incompatible with the conventional **Cu**-based **underbump metallization** (UBM) schemes. The tin in the solder reacts with the **copper layer** of the UBM, depleting the UBM of copper and causing loss of adhesion and a weak interface. Use of new **under bump** metallization schemes with Ni or CuNi alloys as the solderable layer were investigated in this study. Instead of Cr, a Ti-based adhesion layer was used to decrease the amount of stress in the CuNi layer. Flip **chip** solder joints were made in which three Sn-Bi-Ag based lead-free solders were reflowed to several UBM pads of different compositions. The resulting interfacial microstructures were examined by SEM/EDX analysis of cross-sectioned samples. The joints were also mechanically tested in fatigue and shear to assess the quality and reliability of the interface. (Author abstract) 8 Refs.

21/3,AB/17 (Item 1 from file: 34)

DIALOG(R)File 34:SciSearch(R) Cited Ref Sci

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11372537 Genuine Article#: 643PF Number of References: 15

Title: Effects of Ni thickness and reflow times on interfacial reactions between Ni/**Cu under-bump metallization** and eutectic Sn-Pb solder in flip-**chip** technology (ABSTRACT AVAILABLE)

Author(s): Huang CS (REPRINT) ; Duh JG; Chen YM; Wang JH

Corporate Source: Natl Tsing Hua Univ, Dept Mat Sci & Engr, Hsinchu

30043//Taiwan/ (REPRINT); Natl Tsing Hua Univ, Dept Mat Sci &

Engr, Hsinchu 30043//Taiwan/; Natl Chiao Tung Univ, Dept Mat Sci &

Engr, Hsinchu//Taiwan/; Chungwa telecom Co Ltd, Taoyuan//Taiwan/

Journal: JOURNAL OF ELECTRONIC MATERIALS, 2003, V32, N2 (FEB), P89-94

ISSN: 0361-5235 Publication date: 20030200

Publisher: MINERALS METALS MATERIALS SOC, 184 THORN HILL RD, WARRENDALE, PA 15086 USA

Language: English Document Type: ARTICLE

Abstract: Flip-**chip** interconnection technology plays a key role in today's electronics packaging. Understanding the interfacial reactions between the solder and **under-bump metallization** (UBM) is, thus, essential. In this study, different thicknesses of electroplated Ni were used to evaluate the phase transformation between Ni/**Cu under-bump** metallurgy and eutectic Sn-Pb solder in the 63Sn-37Pb/Ni/Cu/Ti/Si₃N₄/Si multilayer structure for the flip-**chip** technology. Interfacial reaction products varied with reflow times. After the first reflow, **layered** (Ni_{1-x}Cu_x)(3)Sn-4 was found between solder and Ni. However, there were two interfacial reaction products formed between solders and the UBM after three or more times reflow. The **layered** (Ni_{1-x}Cu_x)(3)Sn-4 was next to the Ni/Cu UBM. The islandlike (Cu_{1-y}Ni_y)(6)Sn-5 was formed between (Ni,Cu)(3)Sn-4 and solders. The amounts of (Cu_{1-y}Ni_y)(6)Sn-5 intermetallic compound (IMC) could be related to the Ni thickness and reflow times. In addition, the influence of Cu contents on phase transformation during reflow was also studied.

21/3,AB/18 (Item 2 from file: 34)
 DIALOG(R)File 34:SciSearch(R) Cited Ref Sci
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11170268 Genuine Article#: 617PA Number of References: 17
 Title: Failure mechanism of lead-free solder joints in flip **chip** packages (ABSTRACT AVAILABLE)
 Author(s): Zhang F (REPRINT) ; Li M; Balakrisnan B; Chen WT
 Corporate Source: Inst Mat Res & Engn,Singapore 117602//Singapore/ (REPRINT); Inst Mat Res & Engn,Singapore 117602//Singapore/; ASE US Inc,Santa Clara//CA/95054
 Journal: JOURNAL OF ELECTRONIC MATERIALS, 2002, V31, N11 (NOV), P1256-1263
 ISSN: 0361-5235 Publication date: 20021100
 Publisher: MINERALS METALS MATERIALS SOC, 184 THORN HILL RD, WARRENDALE, PA 15086 USA
 Language: English Document Type: ARTICLE
 Abstract: The failure mechanisms of SnAgCu solder on Al/Ni(V)/Cu thin-film, **underbump** metallurgy (UBM) were investigated after multiple reflows and hightemperature storage using a ball shear test, fracture-surface analysis, and cross-sectional microstructure examination. The results were also compared with those of eutectic SnPb solder. The Al/Ni (V)/Cu thin-film UBM was found to be robust enough to resist multiple reflows and thermal aging at conditions used for normal production purposes in both SnAgCu and eutectic SnPb systems. It was found that, in the SnAgCu system, the failure mode changed with the number of reflows, relating to the consumption of the thin-film UBM because of the severe interfacial reaction between the solder and the UBM layer. After high-temperature storage, the solder joints failed inside the solder ball in a ductile manner in both SnAgCu and SnPb systems. Very fine Ag3Sn particles were formed during multiple reflows in the SnAgCu system. They were found to be able to strengthen the bulk solder. The dispersion-strengthening effect of Ag3Sn was lost after a short period of thermal aging, caused by the rapid coarsening of these fine particles.

21/3,AB/19 (Item 3 from file: 34)
 DIALOG(R)File 34:SciSearch(R) Cited Ref Sci
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10793414 Genuine Article#: 569KN Number of References: 120
 Title: Six cases of reliability study of Pb-free solder joints in electronic packaging technology (ABSTRACT AVAILABLE)
 Author(s): Zeng K; Tu KN (REPRINT)
 Corporate Source: Univ Calif Los Angeles,Dept Mat Sci & Engn,Los Angeles//CA/90095 (REPRINT); Univ Calif Los Angeles,Dept Mat Sci & Engn,Los Angeles//CA/90095
 Journal: MATERIALS SCIENCE & ENGINEERING R-REPORTS, 2002, V38, N2 (JUN 14), P55-105
 ISSN: 0927-796X Publication date: 20020614
 Publisher: ELSEVIER SCIENCE SA, PO BOX 564, 1001 LAUSANNE, SWITZERLAND
 Language: English Document Type: REVIEW
 Abstract: Solder is widely used to connect **chips** to their packaging substrates in flip **chip** technology as well as in P surface mount technology. At present, the electronic packaging industry is actively searching for Pb-free solders due to environmental concern of Pb-based solders. Concerning the reliability of Pb-free solders, some electronic companies are reluctant to adopt them into their high-end products. Hence, a review of the reliability behavior of Pb-free solders is timely. We use the format of "case study" to review six reliability problems of Pb-free solders in electronic packaging technology. We

conducted analysis of these cases on the basis of thermodynamic driving force, time-dependent kinetic processes, and morphology and microstructure changes. We made a direct comparison to the similar problem in SnPb solder whenever it is available. Specifically, we reviewed: (1) interfacial reactions between Pb-free solder and thick metallization of bond-pad on the substrate-side, (2) interfacial reactions between Pb-free solder and thin-film **under-bump** metallization on the **chip**-side, (3) the growth of a layered intermetallic compound (IMC) by ripening in solid state aging of solder joints, (4) a long range interaction between **chip**-side and substrate-side metallizations across a solder joint, (5) electromigration in flip **chip** solder joints, and finally (6) Sn whisker growth on Pb-free finish on Cu leadframe. Perhaps, these cases may serve as helpful references to the understanding of other reliability behaviors of Pb-free solders. (C) 2002 Published by Elsevier Science B.V.

21/3,AB/20 (Item 4 from file: 34)
 DIALOG(R)File 34:SciSearch(R) Cited Ref Sci
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10007765 Genuine Article#: 474DJ Number of References: 13
 Title: Investigation of interfacial reaction between Sn-Ag eutectic solder and Au/Ni/Cu/Ti thin **film** metallization (ABSTRACT AVAILABLE)
 Author(s): Park JY (REPRINT) ; Yang CW; Ha JS; Kim CU; Kwon EJ; Jung SB; Kang CS
 Corporate Source: Univ Texas,Arlington//TX/76019 (REPRINT); Univ Texas,Arlington//TX/76019; Sung Kyun Kwan Univ,Sch Met & Mat Engrn,Suwon 440746//South Korea/; Seoul Natl Univ,Sch Mat Sci & Engrn,Seoul//South Korea/
 Journal: JOURNAL OF ELECTRONIC MATERIALS, 2001, V30, N9 (SEP), P1165-1170
 ISSN: 0361-5235 Publication date: 20010900
 Publisher: MINERALS METALS MATERIALS SOC, 184 THORN HILL RD, WARRENDALE, PA 15086 USA

Language: English Document Type: ARTICLE

Abstract: This paper reports the formation of intermetallic compounds in Au/Ni/Cu/Ti **under-bump**-metallization (UBM) structure reacted with Ag-Sn eutectic solder. In this study, UBM is prepared by evaporating Au(500 Angstrom)/Ni(1000 Angstrom)/Cu(7500 Angstrom)/Ti(700 Angstrom) thin films on top of Si substrates. It is then reacted with Ag-Sn eutectic solder at 260 degreesC for various times to induce different stages of the interfacial reaction. Microstructural examination of the interface, using both chemical and crystallographic analysis, indicates that two types of intermetallic compounds are formed during the interfacial reaction. The first phase, formed at the initial stage of the reaction, is predominantly Ni₃Sn₄. At longer times the Ni₃Sn₄ phase transforms into (Cu,Ni)₆Sn₅, probably induced by interdiffusion of Cu and Ni. At this stage, the underlying **Cu layer** also reacts with Sn and forms the same phase, (Cu,Ni)₆Sn₅. As a result, the fully reacted interface is found to consist of two intermetallic layers with the same phase but different morphologies.

21/3,AB/21 (Item 5 from file: 34)
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09962499 Genuine Article#: 470QB Number of References: 22

Title: Development of an etchant for selectively etching TiW_Nx in the presence of electroplated 95%Pb-5%Sn solder (ABSTRACT AVAILABLE)
 Author(s): Ramanathan LN (REPRINT) ; Mitchell D
 Corporate Source: Motorola Inc, Final Mfg Technol Ctr, Chandler//AZ/85224 (REPRINT); Motorola Inc, Final Mfg Technol Ctr, Chandler//AZ/85224
 Journal: IEEE TRANSACTIONS ON COMPONENTS AND PACKAGING TECHNOLOGIES, 2001, V24, N3 (SEP), P425-430
 ISSN: 1521-3331 Publication date: 20010900
 Publisher: IEEE-INST ELECTRICAL ELECTRONICS ENGINEERS INC, 345 E 47TH ST, NEW YORK, NY 10017-2394 USA
 Language: English Document Type: ARTICLE

Abstract: Shrinking **die** sizes and increasing I/O density is motivating the push toward flip **chip** packages. A flip **chip** interconnection system with a **under bump** metallurgy stack containing sputtered TiW_Nx/sputtered **Cu**/electroplated **Cu stud**/electroplated 95%Pb-5%Sn was developed. An important step in the above process is the selective etching of the sputtered **Cu bus layer** and the TiW_Nx barrier layer, in the presence of the Pb-Sn solder. The **Cu bus layer** was selectively etched using commercial etchants. However, no commercial etchants were available for selectively etching the TiW_Nx layer. H₂O₂-NH₄ OH based etching systems, popularly known as Standard Clean-1 cleaning solutions, have been extensively used to clean silicon **wafers** in front end **wafer** fabrication where only trace metal contamination exists. Since metals like lead, copper, titanium, tin and tungsten catalyze the heterogeneous decomposition of the peroxide, the unstable H₂O₂-NH₄ OH based etching systems are rarely used to etch metal films. In this paper the development of a H₂O₂-NH₄ OH based etchant to selectively etch the sputtered TiW_Nx films in the presence of electroplated 95%Pb-5%Sn solder bumps is discussed. A 2(3) full factorial experiment with mid point was conducted to establish the etchant composition, as well as process temperature, that give satisfactory responses with respect to etch time, permissible undercut of the **Cu stud** (caused by the NH₄ OH), and acceptable bump shape after reflow. Statistical analysis was used to understand the significant factors influencing the etch rate and undercut. An etchant containing 6% by volume of 30%-H₂O₂ and 0.75% by volume of 30%-NH₄ OH operated at a temperature of 37 degreesC was found to give satisfactory results.

21/3, AB/22 (Item 1 from file: 35)
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01733865 AADAAI9961671
 Reliability of solder joints in flip **chip** technology: Approaches from fundamental studies of wetting, interfacial reaction, mechanical shear testing, and electromigration
 Author: Liu, Cheng-Yi
 Degree: Ph.D.
 Year: 2000
 Corporate Source/Institution: University of California, Los Angeles (0031)
 Source: VOLUME 61/02-B OF DISSERTATION ABSTRACTS INTERNATIONAL.
 PAGE 1033. 102 PAGES

In the first part of this work, the mechanism of the reactive wetting is studied by using the system of molten SnPb solders on Cu. We conclude that the driving force of reactive wetting must take into account, besides the capillary force, the free energy change due to intermetallic compound formation.

In the second part, we tested flip-chip solder bonded Si samples under tensile and shear loading as a function of annealing time. The solder bump was eutectic SnPb and the under-bump thin film metallization was Cu/Cr deposited on oxidized Si. We found that the failure mode is interfacial fracture and the fracture strength decreases rapidly with annealing time. From SEM observations, the fracture occurs at Cu-Sn/Cr interface. We conclude that it is the metallurgical reaction that has brought the solder into direct contact with Cr surface. The weak joint is due to the spalling of Cu-Sn compound grains from the Cr surface, especially near the edges and corners of the joint. To solve the spalling problem, trilayer Cu/Ni(V)/Al thin film metallization is a potential candidate, beside thick Ni(p) UBM. We have used cross-sectional SEM and TEM to study the wetting reaction between molten eutectic SnPb solder and a sputtered trilayer Cu/Ni(V)/Al thin film metallization. No spalling has been observed. This result indicates that the Cu/Ni(V)/Al or $\text{Cu}_{60}\text{Sn}_{40}/\text{Ni(V)/Al}$ is a stable thin film metallization for the low temperature eutectic SnPb solder.

In the third part, using thin film strips, we have investigated electromigration of six different compositions of SnPb solders at current density of 10^5 Amp/cm² near ambient temperature. The six compositions are pure Sn, $\text{Sn}_{80}\text{Pb}_{20}$, $\text{Sn}_{70}\text{Pb}_{30}$, $\text{Sn}_{63}\text{Pb}_{37}$ (eutectic), $\text{Sn}_{40}\text{Pb}_{60}$, and $\text{Sn}_{5}\text{Pb}_{95}$. The eutectic alloy, with the lowest melting point and a high density of interfaces, was found to have the fastest hillock growth. As composition moving toward the two terminal phases, the hillock growth rate decreases because the volume fractions of primary phases having more EM resistance increase. But it increases again in the pure Sn. Being the kinetic path of mass transport, the interface between Sn and Pb also serves as the place to initiate hillock and void formation. Besides hillock and void formation, electromigration has induced a substantial microstructural change in the two-phase alloy, e.g., there is a large amount of grain growth of the Pb phase in the eutectic alloy.

24/3,AB/1 (Item 1 from file: 2)
 DIALOG(R)File 2:INSPEC
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7011962 INSPEC Abstract Number: B2001-09-2550F-089

Title: Organic solution deposition of **copper seed layers** onto barrier metals

Author(s): Gu, H.; Fang, R.; O'Keefe, T.J.; O'Keefe, M.J.; Shih, W.-S.; Snook, J.A.M.; Leedy, K.D.; Cortez, R.

Author Affiliation: Dept. of Metall. Eng., Missouri Univ., Rolla, MO, USA

Conference Title: Materials, Technology and Reliability for Advanced Interconnects and Low-k Dielectrics. Symposium (Materials Research Society Symposium Proceedings Vol.612) p.1-6

Editor(s): Oehrlein, G.S.; Maex, K.; Joo, Y.-C.; Ogawa, S.; Wetzels, J.T.

Publisher: Mater. Res. Soc, Warrendale, PA, USA

Publication Date: 2001 Country of Publication: USA xvii+592 pp.

ISBN: 1 55899 520 X Material Identity Number: XX-2001-01159

Conference Title: Materials, Technology and Reliability for Advanced Interconnects and Low-k Dielectrics. Symposium

Conference Date: 23-27 April 2000 Conference Location: San Francisco, CA, USA

Language: English

Abstract: Spontaneous deposition of **copper seed layers** from metal bearing organic based solutions on to sputter deposited titanium, titanium nitride, and tantalum diffusion barrier thin films has been demonstrated. Based on electrochemically driven cementation exchange reactions, the process was used to produce adherent, selectively deposited **copper metal particulate films** on blanket and patterned barrier metal thin films on silicon substrates. The organic solution deposited **copper films** were capable of acting as seed layers for subsequent electrolytic and electroless copper deposition processes using standard plating baths. Electroless and electrolytic **copper films** from 0.1 μm to 1.0 μm thick were produced on a variety of samples on which the organic solution copper acted as the initial catalytic seed layer. The feasibility of using organic solution deposited palladium as a seed layer followed by electroless copper deposition has also been demonstrated. In addition, experiments conducted on patterned barrier metal samples with exposed areas of dielectric such as **polyimide** indicated that no organic solution copper or palladium deposition occurred on the insulating materials.

Subfile: B

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24/3,AB/2 (Item 2 from file: 2)
 DIALOG(R)File 2:INSPEC
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6595802 INSPEC Abstract Number: B2000-06-2550F-085

Title: The effect of **polyimide** passivation on the electromigration in sputtered **copper films**

Author(s): Jiann-Shan Jiang; Bi-Shiou Chiou

Author Affiliation: Dept. of Electron. Eng., Nat. Chiao Tung Univ., Hsinchu, Taiwan

Journal: International Journal of Microcircuits and Electronic Packaging vol.22, no.4 p.395-400

Publisher: IMAPS-Int. Microelectron. & Packaging Soc,

Publication Date: 1999 Country of Publication: USA

CODEN: IMEPE5 ISSN: 1063-1674

SICI: 1063-1674(1999)22:4L.395:EPPE;1-7

Material Identity Number: P802-2000-002

Language: English

Abstract: Electromigration damage (EMD) is one of the major causes of failures in interconnects. In this study, the electromigration of Cu with **polyimide** passivation is investigated with an isothermal resistance change method. Resistance decreases in passivated samples caused by diffusion of **polyimide** ingredients into copper are observed. **Polyimide** passivation enhances the lifetime of the Cu metallization (sputtered **copper films**). The lifetime of passivated **copper films** are from 1.1 to 29 times those of the unpassivated films, depending on the passivation materials and the current stressing conditions. The rigidity of the passivation is found to be the controlling factor for lifetime improvement. In addition, the adhesion strength between the Cu and the **polyimide** is studied in this work. A TiWN interlayer promotes adhesion between the Cu and the **polyimide** interface.

Subfile: B

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24/3,AB/3 (Item 3 from file: 2)

DIALOG(R)File 2:INSPEC

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6519925 INSPEC Abstract Number: B2000-04-0170J-043

Title: Flexible laminate substrates for **chip** scale packaging applications

Author(s): Bergstresser, T.; Sallo, J.S.

Author Affiliation: Gould Electron., Eastlake, OH, USA

Conference Title: IPC Chip Scale and BGA National Symposium. Proceedings. Pursuit of the Perfect Package Part vol.1 p.143-9 vol.1

Publisher: IPC, Northbrook, IL, USA

Publication Date: 1998 Country of Publication: USA 2 vol. 183+96 pp.

Material Identity Number: XX-1999-01212

Conference Title: Proceedings of IPC Chip Scale/Ball Grid Array National Symposium

Conference Date: 6-7 May 1999 Conference Location: Santa Clara, CA, USA

Language: English

Abstract: **Chip** scale packaging has become an increasingly popular packaging technology for applications requiring small size, light weight, and high density interconnections. One CSP technology involves the use of flexible laminate materials to provide one or more layers of interconnection. In this paper, characteristics and performance of adhesiveless flexible laminate substrates for CSP applications are discussed. A typical substrate construction includes a **polyimide** substrate, a thin vacuum deposited **metal** tiecoat, a **copper** seedcoat, and an electrodeposited **copper layer**. One or both sides of the **polyimide** substrate may be **metallized**, and the **copper layers** may be of different thicknesses. Certain copper properties may be developed to better withstand mechanical and thermal stresses imposed during processing or finished device operation. Very thin copper can be provided in order to facilitate the required fine conductor lines and spacing. One key performance requirement derives from CSP reliability tests involving exposure to elevated humidity and temperature. Under such conditions, adhesion between **polyimide** substrate and metallization layers can deteriorate. Selection of an appropriate metal tiecoat and **polyimide** substrate can minimize adhesion losses and provide acceptable reliability. Other performance attributes discussed include good copper fatigue behaviour and dimensional stability.

Subfile: B

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24/3,AB/4 (Item 4 from file: 2)
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6358789 INSPEC Abstract Number: B1999-10-2250-011

Title: **Benzocyclobutene/copper** reliability **study**

Author(s): Yasmeen, N.; Maner, K.; Ang, S.S.; Porter, E.V.; Ulrich, R.K.; Brown, W.D.

Author Affiliation: High Density Electron. Center, Arkansas Univ., Fayetteville, AR, USA

Conference Title: Dielectric Material Integration for Microelectronics
 p.299-312

Editor(s): Brown, W.D.; Ang, S.S.; Loboda, M.; Sammakia, B.; Singh, R.; Rathore, H.S.

Publisher: Electrochem. Soc, Pennington, NJ, USA

Publication Date: 1998 Country of Publication: USA x+366 pp.

ISBN: 1 56677 197 8 Material Identity Number: XX-1999-02023

Conference Title: Dielectric Material Integration for Microelectronics

Conference Date: 3-8 May 1998 Conference Location: San Diego, CA, USA

Language: English

Abstract: Due to the attractiveness of the **benzocyclobutene** (BCB)/copper system for application to electronic packaging, the adhesion of BCB to copper and copper to BCB was investigated. Delamination of BCB from copper was observed to occur during electroless nickel plating on copper when aminopropyltriethoxysilane (APS) was used as an adhesion promoter. No delamination occurred when an adhesion promoter consisting of 1-methoxy-2-propanol (1M2P) was used. Delamination was observed during pull testing conducted following 100 thermal shock cycles of samples with APS adhesion promoter. Samples fabricated with two BCB and two **copper layers** using the 1M2P promoter adhered better than samples with APS following 100 cycles of thermal shock. All BCB delaminations during pull testing occurred at a strength greater than 6.9 MPa which is considered to be acceptable for MCM applications.

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24/3,AB/5 (Item 5 from file: 2)
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6083412 INSPEC Abstract Number: B9812-0170J-085

Title: High performance thin film single **chip** module

Author(s): Giri, A.; Kamath, S.; O'Connor, D.; Langenthal, S.; Perfecto, E.; Pennacchia, J.

Author Affiliation: Div. of Microelectron., IBM Corp., Hopewell Junction, NY, USA

Conference Title: 1998 Proceedings. 48th Electronic Components and Technology Conference (Cat. No.98CH36206) p.674-9

Publisher: IEEE, New York, NY, USA

Publication Date: 1998 Country of Publication: USA xxv+1476 pp.

ISBN: 0 7803 4526 6 Material Identity Number: XX98-01334

U.S. Copyright Clearance Center Code: 0 7803 4526 6/98/\$10.00

Conference Title: 1998 Proceedings. 48th Electronic Components and Technology Conference (Cat. No.98CH36206)

Conference Sponsor: IEEE Components, Packaging & Manuf. Technol. Soc.; Electron. Ind. Assoc

Conference Date: 25-28 May 1998 Conference Location: Seattle, WA, USA

Language: English

Abstract: This paper describes development of high density single **chip** modules for flip **chip** (C4) area array interconnect such that the electrical, thermal, and reliability needs are met through an optimal tradeoff between system performance and module cost. Prototype test vehicles were designed and built using co-fired alumina as the base carrier for two levels of **copper-polyimide** thin films containing the bulk of signal wiring and a power/ground plane. Flip-**chip die** with high melt bumps were joined directly to **copper pads** on the thin film substrate. Reliability aspects of this interconnect product, such as, wettability and joinability characteristics of thin **Cu films** and integrity of thin film via interconnections as a function of pre- and post-thermal cycling have been studied. Also, module encapsulation aspects are briefly discussed. The results of electrical characterization of the thin film substrate prior to **die** attach, as well as simultaneous switching noise measurements with a functional test **die** are presented.

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24/3,AB/6 (Item 6 from file: 2)
DIALOG(R)File 2:INSPEC
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5851459 INSPEC Abstract Number: B9804-0170J-053

Title: A low-cost, flexible ball-grid-array multichip module technology

Author(s): Ang, S.S.; Arnn, D.A.; Meyer, D.J.; Schaper, L.W.; Brown, W.D.

Author Affiliation: Dept. of Electr. Eng., Arkansas Univ., Fayetteville, AR, USA

Journal: Proceedings of the SPIE - The International Society for Optical Engineering Conference Title: Proc. SPIE - Int. Soc. Opt. Eng. (USA)
vol.3184 p.13-21

Publisher: SPIE-Int. Soc. Opt. Eng,

Publication Date: 1997 Country of Publication: USA

CODEN: PSISDG ISSN: 0277-786X

SICI: 0277-786X(1997)3184L:13:CFBG;1-H

Material Identity Number: C574-97226

U.S. Copyright Clearance Center Code: 0277-786X/97/\$10.00

Conference Title: Microelectronic Packaging and Laser Processing

Conference Sponsor: SPIE; SPIE Singapore Chapter; Inst. Phys. Singapore; et al

Conference Date: 25-26 June 1997 Conference Location: Singapore

Language: English

Abstract: In pursuit of lower cost packaging solutions, a new type of multichip module (MCM) has been designed and fabricated on flexible **polyimide** films. The new technology uses a double-sided **copper metallized**, through-via, **polyimide** film with a ball-grid-array packaging solution. The enabling technology is the interconnected Mesh Power System (IMPS) MCM topology which provides the signal wiring and power distribution using two metal layers instead of the four-layer metallization scheme as in conventional MCM topology. One side of the **polyimide** film has passivation openings where **chips** are wire bonded; this side is encapsulated to provide structural rigidity and environmental protection of the circuitry. The other side has solder pads where solder spheres are placed and reflowed to create a ball grid array package. The two-layer **copper metallized polyimide film** substrates with through-vias are obtained either by laser drilling of pre-metallized **polyimide** films or reactive ion etching of **polyimide** layers fabricated from their liquid precursor. Issues of solder joint integrity and module warp were investigated. Preliminary test

data are also reported.

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24/3,AB/7 (Item 7 from file: 2)
DIALOG(R)File 2:INSPEC
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5590839 INSPEC Abstract Number: B9707-0170J-030

Title: Interfacial **layer** properties of **copper-sputtered polyimide films**

Author(s): Toth, J.J.; Hirt, A.M.

Author Affiliation: Pacific Northwest Lab., Richland, WA, USA

Conference Title: Proceedings of the 1996 International Electronics Packaging Conference p.477-82

Publisher: Int. Electron. Packaging Soc, Edina, MN, USA

Publication Date: 1996 Country of Publication: USA 681 pp.

Material Identity Number: XX96-02693

Conference Title: Proceedings of International Electronics Packaging Society Conference. 1996 Annual Conference 'A Powerhouse Program'

Conference Sponsor: Int. Electron. Packaging Soc

Conference Date: 29 Sept.-1 Oct. 1996 Conference Location: Austin, TX, USA

Language: English

Abstract: This paper gives results of material characterizations of interfacial **layers** of **Cu-sputtered polyimide films** built up by electroplating for MCM and flex circuit packages. **Cu-sputtered polyimide film** is adhesiveless and is an intermediate material for high density circuit packaging. The intermediate transition **metal layers** between **Cu** and **polyimide** have been used to improve **Cu layer** adhesion, allowing electrolytic layers to be easily built up over the sputtered layer during fabrication. However, metal oxide layers are now used in addition to transition metal layers as the intermediate layer. **Polyimide** base laminate MCM-L material used in typical high density packaging designs was used here. **Cu** sputtered thickness was ≤ 5000 AA. **Cu** was also electroplated over the sputtered surface to form a 15 and 35 μm **Cu** foil on the **polyimide**. The **Cu-polyimide** interfacial **layer** was studied. XPS surface analysis testing was customized to avoid interpretive problems with interfacial layer identification. XPS analysis was combined with material removal by ion beam etching to investigate compositional variation with depth. High resolution spectra were collected from the **Cu** surface facing the **polyimide**. The data summarized indicates the presence of oxides extending into the **Cu** to about 300 AA before disappearing. The oxide is in the form of both **Cu/sub 2/O** and **CuO** phases. The oxide layer is considerable, so that the existence of this interfacial layer and its effects are not negligible. The presence of the oxides in the **Cu-sputtered polyimide film** contribute to the excellent manufacturability and material characteristics of current base materials.

Subfile: B

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24/3,AB/8 (Item 8 from file: 2)
DIALOG(R)File 2:INSPEC
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5471337 INSPEC Abstract Number: B9702-2250-018

Title: Thin film laminated multilayer wiring substrate

Author(s): Takahashi, A.; Itabashi, T.; Watanabe, R.; Miwa, T.; Akahoshi, H.

Author Affiliation: Hitach Ltd., Japan

Conference Title: Proceedings of the Printed Circuit World Convention VII p.P13/1-10

Publisher: Printed Circuit World Convention, Basel, Switzerland

Publication Date: 1996 Country of Publication: Switzerland 1056 pp.

Material Identity Number: XX96-00764

Conference Title: Proceedings of the Printed Circuit World Convention VII

Conference Sponsor: EIPC; IPC; PCIF; IMF; JPCA

Conference Date: 21-24 May 1996 Conference Location: Basel, Switzerland

Language: English

Abstract: The use of MCM packaging, which has an important role in realizing high performance mainframe computers, is growing and extending into the fields of high performance workstations and telecommunications. MCM technologies can be classified into three categories: MCM-C (co-fired ceramic), MCM-L (laminate) and MCM-D (deposited dielectric). MCM-D substrates provide the highest resolution and greatest overall routability. Several fabrication processes for thin film multilayer structures have been reported, based on spin-coating of **polyimide** with repeated heating to evaporate solvents and cure the material. These processes are rather complex and time consuming. In addition, planarization and getting a pin-hole free **polyimide** layer are crucial issues for building up several layers. To overcome these problems and realize high reliability, a new fabrication process for thin film multilayer wiring has been proposed. The process has high productivity and involves laminating a high temperature resistant polymer film with an adhesive layer, via hole formation by plasma etching and filling by electroless **Cu metallization**. A high heat resistant adhesive with **polyimide** matrix and bismaleimide melting constituent was produced. This adhesive showed enough fluidity for void-free lamination and had excellent heat resistance to >330 degrees C after curing. Forming technologies for microvia holes of $\Phi = 25 \mu m$ were established by a conformal mask method using plasma etch and metallization processes to fill the via-holes by electroless **Cu** plating. Thin **film** multilayer substrates with six conductive layers, including two signal layers, were obtained.

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24/3,AB/9 (Item 9 from file: 2)

DIALOG(R)File 2:INSPEC

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04336567 INSPEC Abstract Number: B9303-2220J-002

Title: Electrical characterization of a reduced **metal layer copper-polyimide** multi-**chip** module substrate

Author(s): Hashemi, H.; Sommerfeldt, S.; Miracky, R.

Author Affiliation: Microelectronics & Computer Technol. Corp., Austin, TX, USA

Conference Title: IEPS. Proceedings of the Technical Conference. 1992 International Electronics Packaging Conference p.351-65 vol.1

Publisher: Int. Electron. Packaging Soc, Wheaton, IL, USA

Publication Date: 1992 Country of Publication: USA 2 vol. 1185 pp.

Conference Date: 27-30 Sept. 1992 Conference Location: Austin, TX, USA

Language: English

Abstract: The authors have characterized the electrical properties of a three-metal-layer thin-film **copper-polyimide** multi-**chip** module (MCM) substrate. This substrate is unlike the typical MCM substrate in that it consists of two underlying wiring layers which are

design-independent, and a third, top level of interconnect which contains the design-specific features and bonding sites. Additionally, the design requires fewer interconnect layers because ground wires are distributed among the same layers as the signal interconnects. Hence, such MCMs are considerably lower cost. They conclude from this analysis that this semi-custom, reduced layer MCM design has electrical performance similar to custom MCMs, being able to meet 100 MHz, 1 nanosecond rise-time applications. Furthermore, it has low power supply impedance (approximately $=4 \Omega$) and low crosstalk ($<10\%$). There are two major trade-offs. The first trade-off arises from the semi-custom nature of the design. There is a slight increase in the total series resistance of a given interconnect net due to slightly longer path length. The second trade-off arises from the reduced-layer character of the design. The propagation delay is increased by roughly 30% compared to the case in which there is a ground plane. The bandwidth can be extended to accommodate 200 to 300 MHz clock rate digital systems.

Subfile: B

24/3,AB/10 (Item 10 from file: 2)

DIALOG(R)File 2:INSPEC

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04112115 INSPEC Abstract Number: B9204-2220J-045

Title: Advances in MCM fabrication with **benzocyclobutene** dielectric

Author(s): Heistand, R.H.; DeVellis, R.; Manial, T.A.; Kennedy, A.P.; Garrou, P.E.; Stokich, T.M.; Townsend, P.H.; Adema, G.M.; Berry, M.J.; Turlik, I.

Author Affiliation: Dow Chem. Co., Midland, MI, USA

Conference Title: ISHM '91 Proceedings. Proceedings of the 1991 International Symposium on Microelectronics p.96-100

Publisher: Int. Soc. Hybrid Microelectron, Reston, VA, USA

Publication Date: 1991 Country of Publication: USA 555 pp.

Conference Sponsor: Int. Soc. Hybrid Microelectron

Conference Date: 21-23 Oct. 1991 Conference Location: Orlando, FL, USA

Language: English

Abstract: Advances have been made in understanding the processing of the **benzocyclobutene** (DVS-bisBCB) with respect to the effect of cure management on film stress and film adhesion to various interfaces. A 12 **layer** structure (6 **Cu/6 BCB**) has been fabricated on a **Cu/Mo/Cu** substrate without **metal tie layers**. **Copper** migration is not observed in the TEM of a BCB/Cu interface after exposure to 1000 hr at 85 degrees C/85% RH. Adhesion to gold currently utilizes a dual adhesion promoter system. Processes are now in place to achieve excellent adhesion of BCB to metals, ceramics and itself.

Subfile: B

24/3,AB/11 (Item 11 from file: 2)

DIALOG(R)File 2:INSPEC

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03823965 INSPEC Abstract Number: A91036181

Title: Studies of the interface between the epoxy molding compound and the copper leadframe by X-ray photoelectron spectroscopy, Auger electron spectroscopy, and secondary electron microscopy

Author(s): Pan, F.M.; Horng, S.R.; Yang, T.D.; Tang, V.

Author Affiliation: Mater. Res. Lab., Ind. Technol. & Res. Inst., Hsingchu, Taiwan

Journal: Journal of Vacuum Science & Technology A (Vacuum, Surfaces, and Films) vol.8, no.6 p.4074-8

Publication Date: Nov.-Dec. 1990 Country of Publication: USA
 CODEN: JVTAD6 ISSN: 0734-2101
 U.S. Copyright Clearance Center Code: 0734-2101/90/064074-05\$01.00
 Language: English

Abstract: The interface between the epoxy molding compound and the copper leadframe of an **integrated circuit** device has been studied by X-ray photoemission spectroscopy (XPS), Auger electron spectroscopy (AES), and secondary electron microscopy (SEM). The surface of the copper frame is composed of CuO, Cu/sub 2/O, or Cu(OH)/sub 2/ depending on the heat treatment for the frame before the molding process. The XPS results show that cuprous oxide is the primary copper oxide at the interface between the leadframe and the epoxy **polymeric** encapsulant after the molding process. Copper was found to migrate into the epoxy resin side. According to AES and SEM, silica fillers in the molding compound abrade the **copper oxide layer** on the **copper** frame during the molding process, and the injected hot epoxy fluid can interact directly with the freshly exposed **metallic copper** surface. The accelerator of the molding compound segregates to the abraded area at the interface. The rugged interfacial structure close to the molding compound injection gate is probably the primary factor causing the microgap problem at the interface.

Subfile: A

24/3,AB/12 (Item 12 from file: 2)
 DIALOG(R)File 2:INSPEC
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03412063 INSPEC Abstract Number: B89046905

Title: A **copper/polyimide metal**-base packaging technology

Author(s): Takasago, H.; Adachi, K.; Takada, M.

Author Affiliation: Mater. & Electron. Devices Lab., Mitsubishi Electric Corp., Hyogo, Japan

Journal: Journal of Electronic Materials vol.18, no.2, pt.2 p. 319-26

Publication Date: March 1989 Country of Publication: USA

CODEN: JECMA5 ISSN: 0361-5235

Language: English

Abstract: A unique substrate MCPM (Mitsubishi **Copper Polyimide Metal**-base) technology has been developed by applying basic copper/**polyimide** technology. This new substrate technology MCPM is suited for a high-density, multi-layer, multi-**chip**, high-power module/package, such as used for a computer. The new MCPM was processed using a **copper metal** base (110*110 mm), full **copper** system (all layers) with 50- μ m fine lines. As for pad metallizations for the IC assembly, the authors evaluated both Ni/Au for **chip** and wire ICs and solder for TAB ICs. The total number of assembled ICs is 25. To improve the thermal dispersion, copper thermal vias are simultaneously formed by electro-plating. This thermal via is located between the IC **chip** and **copper metal** base, and promotes heat dispersion. The authors employed one large thermal via (4.5 mm/sup phi /) and four small vias (1.0 mm/sup phi /) for each IC pad. The effect of thermal vias and/or base metal is simulated by a computer analysis and compared with an alumina base substrate. The results show that the thermal vias are effective at lowering the temperature difference between the IC and base substrate, and also lowering the temperature rise of the IC **chip**.

Subfile: B

24/3,AB/13 (Item 13 from file: 2)
 DIALOG(R)File 2:INSPEC
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03038673 INSPEC Abstract Number: B88001282
 Title: Surface mounting on ATFT-hybrid circuits
 Author(s): Krokoszinski, H.-J.; Haas, H.; Oetzmann, H.; Disselnkötter, R.
 Author Affiliation: Central Res. Lab., Brown, Boveri & Cie, Heidelberg, West Germany
 Conference Title: Sixth European Microelectronics Conference Proceedings. ISHM Europe '87 p.361-71
 Publisher: Int. Soc. Hybrid Microelectron. Eur, London, UK
 Publication Date: 1987 Country of Publication: UK 494 pp.
 Conference Date: 3-5 June 1987 Conference Location: Bournemouth, UK
 Language: English
 Abstract: The additive thin film technology (ATFT) has recently been shown to be a versatile means to fabricate complete hybrid circuits including RC-networks, crossing connections and SMD-landing areas. The anorganic-organic passivation layer sequence developed for protection of the network against oxidation and corrosion has now been improved by using a photosensitive **polyimide** layer. The authors investigate electroless plating with Sn, Ni and Au for different evaporated **pad** materials (Cu, Ni). Soldering experiments are carried out on three **pad layer** sequences: Cu/electroless Sn and Cu/ or Ni/electroless Au on glazed ceramic substrates. Cu/Sn-**pad layers** showed perfect wetting behaviour and high bond strength after vapour phase soldering and IR-reflow soldering. Ni/Au-layers, however, seem to be more suitable for bonding purposes.
 Subfile: B

24/3,AB/14 (Item 14 from file: 2)
 DIALOG(R)File 2:INSPEC
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03037708 INSPEC Abstract Number: B88001256
 Title: A new hybrid technology: high density thin **film copper/polyimide** multilayer systems
 Author(s): Kompelien, D.; Moravec, T.J.; DeFlumere, M.
 Author Affiliation: Honeywell Phys. Sci. Center, Bloomington, MN, USA
 Conference Title: Proceedings of the 1986 International Symposium on Microelectronics p.749-57
 Publisher: Int. Soc. Hybrid Microelectron, Reston, VA, USA
 Publication Date: 1986 Country of Publication: USA 915 pp.
 Conference Sponsor: Int. Soc. Hybrid Microelectron.
 Conference Date: 6-8 Oct. 1986 Conference Location: Atlanta, GA, USA
 Language: English
 Abstract: The authors present the design and discuss the fabrication of an 18 **chip** high performance custom signal processing hybrid package fabricated with a new nonceramic interconnect system. The interconnection is accomplished by a high density, 4 **metal layer copper** (2 mil lines on 10 mil pitch) and **polyimide** dielectric on a ceramic substrate. Results are presented on the construction and assembly of this hybrid sealed in a 2.4 inch square hermetic flatpack.
 Subfile: B

24/3,AB/15 (Item 15 from file: 2)
 DIALOG(R)File 2:INSPEC
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03019693 INSPEC Abstract Number: B87073932

Title: Multi-**chip** packaging design for VLSI-based systems

Author(s): Bartlett, C.J.; Segelken, J.M.; Teneketges, N.A.

Author Affiliation: AT& Bell Labs., Murray Hill, NJ, USA

Conference Title: 1987 Proceedings of the 37th Electronic Components Conference (Cat. No.87CH2448-9) p.518-25

Publisher: IEEE, New York, NY, USA

Publication Date: 1987 Country of Publication: USA xii+651 pp.

U.S. Copyright Clearance Center Code: 0569-5503/87/0000-0518\$01.00

Conference Sponsor: IEEE; Electron. Ind. Assoc

Conference Date: 11-13 May 1987 Conference Location: Boston, MA, USA

Language: English

Abstract: A packaging technology that will overcome many of the limitations of conventional packaging is described. The described technology has the capability for assembling devices with more than 200 I/Os, a maximum signal lead length <20 cm, a power and ground lead inductance less than 1.0 nH, a signal lead capacitance less than 20 pF, and a cooling capability greater than 1 watt/cm². The substrate for the proposed packaging technology is a silicon **wafer**. Power and ground is distributed by means of copper planes on either side of the substrate. Two signal layers are positioned above the power plane using the **polyimide** dielectric material. The signal leads are a minimum of 10 μ m wide. With 10 μ m-thick polymer, the line capacitance is approximately 1 pF/cm. **Copper metallization** is used to achieve a resistance of 10 Ω /cm for the minimum-width signal leads with 2 μ m-thick conductors. Devices are attached to the interconnection substrate by means of solder.

Subfile: B

24/3,AB/16 (Item 16 from file: 2)

DIALOG(R)File 2:INSPEC

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02759810 INSPEC Abstract Number: B86062979

Title: Advanced copper/**polyimide** hybrid technology

Author(s): Takasago, H.; Takada, M.; Adachi, K.; Endo, A.; Yamada, K.; Makita, T.; Gofuku, E.; Onishi, Y.

Author Affiliation: Mitsubishi Electr. Corp., Hyogo, Japan

Conference Title: 36th Electronic Components Conference Proceedings 1986 (Cat. No.86CH2302-8) p.481-7

Publisher: IEEE, New York, NY, USA

Publication Date: 1986 Country of Publication: USA 684 pp.

U.S. Copyright Clearance Center Code: 0569-5503/86/0000-0481\$01.00

Conference Sponsor: IEEE; Electron. Ind. Assoc

Conference Date: 5-7 May 1986 Conference Location: Seattle, WA, USA

Language: English

Abstract: A hybrid IC processing approach which combines wet **metallized copper**, air-fired RuO₂/sub 2/ paste, and photoactive **polyimide**, was previously presented by the authors. Now, a further improved new copper/**polyimide** hybrid technology, MCPH (Mitsubishi Copper **Polyimide** Hybrid), has been developed. The MCPH was processed by using a large (106 mm*106 mm) Al/sub 2/O/sub 3/-substrate, full-**copper** system (all **layers**) with 50- μ m fine lines. For the first layer, the full-additive process is adopted, and for other layers, a semi-additive process is used. The photoactive **polyimide** used as interlayer insulation is screen printed, and, after the exposure and development steps, cured in a conveyor belt furnace. This new process has many excellent features, including uniform coating thickness and better mass-production capability. The MCPH fundamental processes are evaluated and established using a test element group having fine lines, small

diameter vias and/or pads, matrix lines, etc.
Subfile: B

24/3,AB/17 (Item 17 from file: 2)
DIALOG(R)File 2:INSPEC
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02340592 INSPEC Abstract Number: B84058655
Title: Fine-line, multilayer hybrids with wet-processed conductors and thick-film resistors
Author(s): Takasago, H.; Takada, M.; Adachi, K.; Endo, A.; Yamada, K.; Onishi, Y.; Morihiro, Y.
Author Affiliation: Mitsubishi Electric Corp., Amagasaki, Japan
Conference Title: 34th Electronic Components Conference p.324-9
Publisher: IEEE, New York, NY, USA
Publication Date: 1984 Country of Publication: USA 537 pp.
U.S. Copyright Clearance Center Code: 0569-5503/84/0000-0324\$01.00
Conference Sponsor: IEEE
Conference Date: 14-16 May 1984 Conference Location: New Orleans, LA, USA

Language: English

Abstract: New processing approaches are developed for high-density hybrids having fine-lining or multilayering and for thick-film resistors. The desirable material combinations of wet-metallized copper, air-fired RuO₂/sub 2/ paste, and photoactive polyimide, are used for the fabrication of two types of hybrids, A and B. Hybrid A utilizes a conventional thick-film circuit having a single conductor layer as a base substrate, and fine lines can easily be attained by the added top copper layer. Hybrid B fully utilizes the advantages of copper for both top and bottom layers. These hybrids are both constructed on alumina based 50*50 mm substrates using the same test circuit patterns, and the chip components are reflow-soldered. When the hybrids are evaluated, both the top and bottom conductors have ample adherence, and the heat-resistance-related features (such as adherence, leaching, appearance, etc.) are also excellent. Thick-film resistors and/or reflowed chip components do not show any negative signs in assembly or various chamber tests. Consequently, the result is the development of a simple and reliable multilayer hybrid process.

Subfile: B

24/3,AB/18 (Item 18 from file: 2)
DIALOG(R)File 2:INSPEC
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01854921 INSPEC Abstract Number: A82052098, B82024842
Title: Protection of M1-Cu surface prior to M2 evaporation (metallised ceramic polyimide technology)
Author(s): Ameen, J.G.; Franchak, N.P.; Nair, K.K.; Smey, S.L.
Author Affiliation: IBM Corp., Armonk, NY, USA
Journal: IBM Technical Disclosure Bulletin vol.24, no.6 p.2989
Publication Date: Nov. 1981 Country of Publication: USA
CODEN: IBMTAA ISSN: 0018-8689
Language: English
Abstract: Describes an oxidation prevention technique for the interconnection between two Cu layers in metallised ceramic polyimide substrate technology. The protective layer, such as naphthalene, vaporises at high temperature in a vacuum leaving a clean surface resulting in a good bond between the vacuum deposited Cu layers.

Subfile: A B

24/3,AB/19 (Item 1 from file: 6)
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1074551 NTIS Accession Number: NTN83-0930

Terminal Strip Facilitates Printed-Circuit Board Changes: Strip can be bonded to board directly over used or unused conductor patterns

(NTIS Tech Note)

National Aeronautics and Space Administration, Washington, DC.

Corp. Source Codes: 011249000

Nov 83 1p

Languages: English

Journal Announcement: GRAI8403

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This citation summarizes a one-page announcement of technology available for utilization. A laminated copper and **polyimide** terminal strip makes it easy to modify a printed-circuit (PC) board, after the board has been fabricated. When epoxied over conductors or an insulating portion of the PC board, the strip provides a series of **solder-coated copper** conductor **pads** to which **integrated-circuit** leads can be soldered for functional changes. The terminal strips can accommodate the leads on a dual, inline IC package or as staggered single or multiple leads on planar mounted flat-packs. With the new terminal strip, changes can be made at any stage of circuit-board population, up until the installation of the board in the final product. Besides serving as a soldering pad for **integrated-circuit** leads, the terminal strip can perform the same function for the circuit-board connector inputs and outputs when their links with board components must be changed. ...FOR ADDITIONAL INFORMATION: Contact: Tech. Transfer Div., P.O. Box 8757, BWI Airport, MD 21240; (301) 621-0100. Refer to: GSC-12748/TN.

24/3,AB/20 (Item 1 from file: 8)
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06120960

E.I. No: EIP02367068360

Title: Advance metallization conference 2001 (AMC 2001)

Author: McKerrow, A.J. (Ed.); Shacham-Diamand, Y. (Ed.); Zaima, S. (Ed.); Ohba, T. (Ed.)

Conference Title: Advanced Metallization Conference 2001 (AMC 2001)

Conference Location: Montreal, Que., Canada Conference Date: 20011008-20011011

E.I. Conference No.: 59475

Source: Advanced Metallization Conference (AMC) 2001. 708p

Publication Year: 2001

CODEN: MRSPDH ISSN: 1048-0854

Language: English

Abstract: The proceedings contains 108 papers from the Advance Metallization Conference 2001 (AMC 2001). The topics discussed include: extending **copper metallization** technology for wiring to end-of-roadmap feature sizes; a Cu interconnect process for the 130 nm process technology node; elimination process-related defects during the fabrication of copper interconnects; integrative aspects of submicron

contact filling by Al-Ge-Cu sputtering and new **wafer** stacking technology for three-dimensional system-on-a-**chip**. (Edited abstract)

24/3,AB/21 (Item 2 from file: 8)
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06117150

E.I. No: EIP02357063585

Title: Photochemical nucleation of copper on **polyimide** surface with 10ns laser irradiation - growth of **copper metal** on **copper** nuclei and deposition of SiO//2 film on **copper metal**

Author: Tokunaga, H.; Ogawa, Y.; Murahara, M.

Corporate Source: Department of Electrical Engineering Tokai University, Hiratuka, Kanagawa 259-1292, Japan

Conference Title: Combinatorial and Artificial Intelligence Methods in Materials Science

Conference Location: Boston, MA, United States Conference Date: 20011126-20011129

E.I. Conference No.: 59423

Source: Materials Research Society Symposium - Proceedings v 700 2002. p 185-191

Publication Year: 2002

CODEN: MRSPDH ISSN: 0272-9172

Language: English

Abstract: Copper atoms were substituted on the **polyimide** surface in the presence of CuSO//4 solution with only 10ns single shot of ArF laser. Then, we have succeeded in substituting copper atoms with dangling bonds of carbon cross-linking with oxygen atoms. In this study, we placed fused silica glass on the **polyimide** surface firstly, and poured the sulfate solution into the gap between the silica glass and the film, forming a thin liquid layer. Then, one shot of circuit patterned ArF laser light having 42mJ/cm**2 was irradiated vertically onto the sample. The photon is dissociating and exciting the copper sulfate solution and the **polyimide** surface. The dissociated copper atoms form the C-O-Cu bond with active oxygen on the **polyimide** surface. However, we were able to grow a **copper** thin film of about 24um after immersing the sample into the electro less plating solution for 15 minutes at 60 degree C. On the other hand, we have laminated transparent and low dielectric constant SiO//2 film at room temperature on **polyimide** surface by using Xe//2* excimer lamp. Then, the **copper** film deposited on **polyimide** film and the piece of silicon were placed in the reaction chamber with mixing NF//3 and O//2 gasses in the ratio of 10:1; total pressure 330 Torr. Then, Xe//2* excimer lamp was irradiated for 20 minutes. By the photochemical reaction, SiF//4 and NO//2 were produced. The adsorbed SiF//4 on copper and **polyimide** surface of one molecule layer and NO//2 up react with NO//2, and growth SiO//2 film on the sample surface. A chain reaction of film formation is brought about by these two chemical reactions of adsorption and oxidization. By this film formation film thickness was not more than 600A after 20 minutes with the Xe//2* excimer lamp irradiation in order to lose in SiF//4. Then, the silicon was supplied, and insufficient SiF//4 was filled by ablating the silicon **wafer** by focused ArF excimer laser beam. 14 Refs.

24/3,AB/22 (Item 3 from file: 8)
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05942302

E.I. No: EIP01476734956

Title: Barrier-~~metal~~-free (BMF), Cu dual-damascene interconnects with Cu-epi-contacts buried in anti-diffusive, low-k organic film

Author: Tada, M.; Ohtake, H.; Harada, Y.; Hiroi, M.; Saito, S.; Onodera, T.; Furutake, N.; Kawahara, J.; Tagami, M.; Kinoshita, K.; Fukai, T.; Mogami, T.; Hayashi, Y.

Corporate Source: System Devices and Fundamental Res. NEC Corporation, Kanagawa 229-1198, Japan

Conference Title: 2001 VLSI Technology Symposium

Conference Location: Kyoto, Japan Conference Date: 20010612-20010614

E.I. Conference No.: 58691

Source: IEEE Symposium on VLSI Circuits, Digest of Technical Papers n TECHNOLOGY SYMP. 2001. p 13-14 (IEEE cat n 01CH37184)

Publication Year: 2001

CODEN: 85PXA5

Language: English

Abstract: Barrier-~~metal~~-free (BMF), Cu dual-damascene interconnects (DDI) are fabricated in the plasma-polymerized, divinyl siloxane bis-**benzocyclobutene** (p-BCB: $k=2.6$) polymer film, which is featured by the anti-diffusive characteristics for the Cu. The BMF-structure has inter-line leak current as low as that of a conventional barrier-inserted structure and is estimated to keep the high insulating property over 10 years under 1MV/cm. The BMF-structure also derives Cu-epi-contacts, reducing the via-resistance of 50% to the conventional Cu/barrier/Cu contacts. The effective dielectric constant was $k_{eff}=3.1$ including very thin SiN etch-stop-layers, accomplishing 20%-fast CMOS device operation to that of the conventional Cu-DDI in the SiO₂ with Ta/TaN barriers. The BMF, Cu-DDIs buried directly in the p-BCB film is one of the ultimate structures for high performance, 0.1μm-CMOS devices and beyond. 4 Refs.

24/3,AB/23 (Item 4 from file: 8)

DIALOG(R)File 8:Ei Compendex(R)

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05561738

E.I. No: EIP00055175490

Title: Development of interposer material (TFI) for face down type CSP

Author: Takahashi, Toyosei

Corporate Source: Sumitomo Bakelite Co, Ltd, Yokohama, Jpn

Conference Title: InterPACK '99: Pacific RIM/ASME International Intersociety Electronics Photonic Packaging Conference 'Advances in Electronic Packaging 1999'

Conference Location: Maui, HI, USA Conference Date: 19990613-19990619

Source: American Society of Mechanical Engineers, EEP v 26 (1) 1999. ASME, USA. p 387-389

Publication Year: 1999

CODEN: EEAEEM

Language: English

Abstract: According to a demand for downsizing of electronic devices, the technology development of miniaturizing semiconductor packages has been made briskly. CSPs appeared in market, and now the amount of CSP is increasing radically. We developed a flexible interposer material (TFI) for face down type CSPs. A package made by using TFI passes through JEDEC Level 3 in a reflow reliability test. TFI consists of three layers, which are a copper foil, a **polyimide** layer, and a **chip** mounting resin layer. The **chip** mounting resin is a polymer material to stick IC **chip** with an interposer, and to protect IC **chip**

from the ambience. The resin has high adhesion force between IC chip and interposer. The resin keeps performance as an adhesive and high peel strength, even if it is heated at 180 degree C before the mounting process. The resin isn't eroded by solvents. The advantages of using TFI are 1) it isn't need for users to coat the resin after the circuit process, 2) the thickness is controlled precisely and uniformly, and 3) it is possible for rigid board makers to manufacture the flexible interposer circuit. (Author abstract) 2 Refs.

24/3,AB/24 (Item 5 from file: 8)
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04819530

E.I. No: EIP97093822049
 Title: Characterizing two metal TBGA
 Author: Gengel, Glenn; Goetz, Martin
 Corporate Source: Sheldahl Micro Products, Longmont, CO, USA
 Conference Title: Proceedings of the 1997 IEEE/CPMT 20th International Electronic Manufacturing Symposium
 Conference Location: Tokyo, Jpn Conference Date: 19970416-19970418
 E.I. Conference No.: 46958
 Source: Proceedings of the IEEE/CPMT International Electronic Manufacturing Technology (IEMT) Symposium 1997. IEEE, Piscataway, NJ, USA, 97CH36059. p 412-415
 Publication Year: 1997
 CODEN: 61UNAI
 Language: English
 Abstract: This paper will characterize two **metal layer copper polyimide** substrate and finished Tape Ball Grid Array (TBGA) semiconductor packages. Data to be presented will cover substrate performance in environmental test, material properties and thermal characterization. Package characterization will address thermal characterization. (Author abstract)

24/3,AB/25 (Item 6 from file: 8)
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04508534

E.I. No: EIP96093343169
 Title: High wiring density **Cu-polyimide** thin film multilayer circuit which is realized by the vertical plated small via
 Author: Matsushima, Naoki; Yamazaki, Tetsuya; Sotokawa, Hideo; Kojima, Hiroyuki; Shigi, Hidetaka
 Corporate Source: Hitachi Ltd, Yokohama, Jpn
 Conference Title: Proceedings of the 1995 IEEE/CPMT 18th International Electronic Manufacturing Technology
 Conference Location: Omiya, Jpn Conference Date: 19951204-19951206
 E.I. Conference No.: 45338
 Source: 1995 Japan IEMT Symposium Proceedings of the IEEE/CPMT International Electronic Manufacturing Technology (IEMT) Symposium 1995. IEEE, Piscataway, NJ, USA, 95CH35994. p 97-100
 Publication Year: 1995
 CODEN: 61UNAI
 Language: English
 Abstract: A fabrication process for **Cu-polyimide** thin film multilayer circuit has been developed for advanced multichip module. The multilayer circuit realizes a high wiring density circuit by

applying **Cu studs** which are deposited by electroplating using the guide patterns made of thick positive photoresist, photosensitive **polyimide** for insulating layers which has low reactivity against Cu, and a polishing process for planarizing dielectric layers. This paper reports on the structure of the circuit and on the thin film process technology. (Author abstract) 5 Refs.

24/3,AB/26 (Item 7 from file: 8)
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04440106

E.I. No: EIP96073239384

Title: Selective seeding of **copper films** on **polyimide**-patterned silicon substrate, using ion implantation

Author: Bhansali, S.; Sood, D.K.

Corporate Source: Royal Melbourne Inst of Technology, Melbourne, Aust

Source: Sensors and Actuators, A: Physical v 52 n 1-3 Mar-Apr 1996. p 126-131

Publication Year: 1996

CODEN: SAAPEB ISSN: 0924-4247

Language: English

Abstract: Our recent work has established the successful use of ion implantation into silicon or **polyimide** to seed the electroless plating of copper on these surfaces. The substrates exhibit a threshold implantation dose, below which the plating does not occur. The threshold implantation dose for seeding of electroless **Cu films** on Pd-implanted Si is observed to be about 20 times lower than that for **polyimide**. In this paper we demonstrate a successful exploitation of this difference in threshold dose to deposit thin copper structures selectively only on (100) silicon **wafer** patterned with **polyimide** and implanted with Pd ions. The process is used to fabricate copper coils with a track width of 10 μ m and track spacing 10 μ m. A detailed analysis of the implanted and plated samples, using Rutherford backscattering spectroscopy (RBS), scanning electron microscopy (SEM) and optical microscopy is also presented. (Author abstract) 6 Refs.

24/3,AB/27 (Item 8 from file: 8)
 DIALOG(R)File 8: Ei Compendex(R)
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04333490

E.I. No: EIP96013010751

Title: Chemical-mechanical polishing of copper with oxide and polymer interlevel dielectrics

Author: Gutmann, Ronald J.; Steigerwald, Joseph M.; You, Lu; Price, David T.; Neiryneck, Jan; Duquette, David J.; Murarka, Shyam P.

Corporate Source: Rensselaer Polytechnic Inst, Troy, NY, USA

Conference Title: Proceedings of the 1995 22nd International Conference on Metallurgical Coatings and Thin Films

Conference Location: San Diego, CA, USA Conference Date: 19950424-19950428

E.I. Conference No.: 44178

Source: Thin Solid Films v 270 n 1-2 Dec 1 1995. p 596-600

Publication Year: 1995

CODEN: THSFAP ISSN: 0040-6090

Language: English

Abstract: Chemical-mechanical polishing (CMP) of copper with oxide interlevel dielectrics has been demonstrated as a viable patterning

approach for copper interconnect structures. This paper summarizes our understanding of the mechanisms involved in copper CMP and presents our results with both oxide interlevel dielectrics (ILDs) and low dielectric constant polymer ILDs. Our two-step model of copper CMP consists of mechanical abrasion of the copper surface followed by removal of the abraded material from the vicinity of the surface and has been developed after extensive electrochemical and CMP experiments with alternative slurries. Although the softer and less process tolerant polymers result in Damascene patterning difficulties compared with oxide ILDs, our results with both **benzocyclobutene** and parylene indicate that manufacturable processes for on-chip interconnect structures can be established with additional fundamental understanding and further development. (Author abstract) 14 Refs.

24/3,AB/28 (Item 9 from file: 8)
 DIALOG(R)File 8: Ei Compendex(R)
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04066547

E.I. No: EIP95022561105

Title: Selective electroless copper plating on silicon seeded by copper ion implantation

Author: Bhansali, S.; Sood, D.K.; Zmood, R.B.

Corporate Source: Royal Melbourne Inst of Technology, Melbourne, Aust

Conference Title: Proceedings of the 21st International Conference on Metallurgical Coatings and Thin Films

Conference Location: San Diego, CA, USA Conference Date: 19940425-19940429

E.I. Conference No.: 42426

Source: Thin Solid Films v 253 n 1-2 Dec 15 1994. p 391-394

Publication Year: 1994

CODEN: THSFAP ISSN: 0040-6090

Language: English

Abstract: We report on the successful use of copper (self) ion implantation into silicon to seed the electroless plating of copper on silicon (100) surfaces. Copper ions were implanted into silicon to doses of 5 multiplied by 10^{14} minus 6.4 multiplied by 10^{16} ions cm^{-2} using a metal vapour vacuum arc ion implanter at extraction voltages of 10 kV and 20 kV. A **copper film** was then deposited onto implanted silicon using a commercial electroless plating solution. The ion energy was kept low enough to facilitate a low critical 'seed' threshold dose which was measured to be 2 multiplied by 10^{15} Cu ions cm^{-2} . Test patterns were made using **polyimide** to study the adaptability of this technique to forming thick structures. Plated films were studied with Rutherford backscattering spectrometry, scanning electron microscopy (SEM), profilometry, energy-dispersive X-rays and Auger electron spectroscopy. The adhesion of films was estimated by a 'Scotch tape test'. The adhesion was found to improve with increasing dose. However, thicker films exhibited rather poor adhesion and high internal stress. Detailed examinations of the top and bottom of the film establish that delamination takes place at the amorphous-crystalline interface of the implanted silicon. SEM results show that the films grow first as isolated islands which become larger and eventually coalesce into a continuous film as the plating time is increased. (Author abstract) 6 Refs.

24/3,AB/29 (Item 10 from file: 8)
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03578645

E.I. Monthly No: EIM9303-018758

Title: Factors affecting the interconnection resistance and yield in the fabrication of multilayer **polyimide**/metal thin film structures.

Author: Shih, D. -Y.; Yeh, H.; Narayan, C.; Lewis, J.; Graham, W.; Nunes, S.; Paraszczak, J.; McGouey, R.; Galligan, E.; Cataldo, J.; Serino, R.; Perfecto, E.; Chang, C. -A.; Deutsch, A.; Rothman, L.; Ritsko, J.; Wilczynski, J.

Conference Title: Proceedings of the 42nd Electronic Components and Technology Conference

Conference Location: San Diego, CA, USA Conference Date: 19920518

E.I. Conference No.: 17724

Source: Proceedings - Electronic Components Conference. Publ by IEEE, IEEE Service Center, Piscataway, NJ, USA (IEEE cat n 92CH3056-9). p 1002-1014

Publication Year: 1992

CODEN: PECCA7 ISSN: 0569-5503 ISBN: 0-8186-2660-7

Language: English

Abstract: The use of a lift-off technique to fabricate a high-density structure consisting of multiple layers of metal/**polyimide** thin film structure on a silicon substrate is described. To achieve better performance and high yield, the authors evaluated the process design, the processing parameters, and the thickness of the Cr/Cu/Cr **metallurgy**, along with the use of suitable **polyimide** dielectrics. The plasma processing conditions, the types of passivation **metals** on Cu, and the use of a siloxane-**polyimide** as the gap-fill/etch-stop material were all shown to play a very critical role in affecting the interconnection resistance and yield of the multilayer thin film structures. By optimizing these parameters the feasibility of fabricating high-density thin film wiring layers with good yield is demonstrated. 21 Refs.

24/3,AB/30 (Item 11 from file: 8)

DIALOG(R)File 8: Ei Compendex(R)

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03076784

E.I. Monthly No: EIM9106-024742

Title: Laser direct writing of copper on various thin-film substrate materials.

Author: Mueller, Heinrich G.; Buschick, Klaus; Schuler, Siegfried; Paredes, Alvaro

Corporate Source: TU Berlin, Berlin, Ger

Conference Title: Proceedings of Symposium E on Surface Processing and Laser Assisted Chemistry of the 1990 E-MRS Spring Conference

Conference Location: Strasbourg, Fr Conference Date: 19900529

E.I. Conference No.: 14478

Source: Applied Surface Science (1985) v 46 n 1-4 Dec-Feb 1990. p 143-147

Publication Year: 1990

CODEN: ASUSEE ISSN: 0169-4332

Language: English

Abstract: Laser direct writing of copper from dried-on **copper** formate **films** on Al//20//3 AIN, **polyimide** and aluminum is described. Writing speeds of 1 to 50 mm/s, and a laser power of up to 2 W have been used. On **polyimide**, argon laser writing is the preferred method. It is shown that multichip module interconnections may be written successfully with this method. (Author abstract) 9 Refs.

24/3,AB/31 (Item 12 from file: 8)

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01504006

E.I. Monthly No: EI8404033145

E.I. Yearly No: EI84063558

Title: ETCHING METHOD FOR Cr/Cu/Al **METALLIZATION**.

Author: Elias, K. L.; Hoffman, H. S.; Sanders, D. R.

Source: IBM Technical Disclosure Bulletin v 25 n 11A Apr 1983 p 5660

Publication Year: 1983

CODEN: IBMTAA ISSN: 0018-8689

Language: ENGLISH

Abstract: One conventional technique for packaging **IC chips** employs a ceramic substrate which has metallization applied to it, and on which metallized substrate the **chips** are mounted by solder connections. It has been proposed that in some cases two levels of metallization may be employed rather than only one. One such system employs a first level of metallization M1 on the ceramic substrate comprised of superimposed **layers** of Cr- Cu-Cr. Over this M1 layer a **polyimide** coating is applied, and then a second layer of metallization M2 is applied over the **polyimide**. The second layer M2 may contain superimposed **layers** of Al-Cu-Cr. It is often desirable to etch through this second layer of metallization.

24/3,AB/32 (Item 1 from file: 34)
 DIALOG(R)File 34:SciSearch(R) Cited Ref Sci
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10563035 Genuine Article#: 542MD Number of References: 35
 Title: Residual stress evolution in dielectric thin films prepared from poly(methylsilsequioxane) precursor (ABSTRACT AVAILABLE)
 Author(s): Oh W; Shin TJ; Ree M (REPRINT) ; Jin MY; Char K
 Corporate Source: Pohang Univ Sci & Technol, Dept Chem, Ctr Integrated Mol Syst, Funct Polymer Thin Film Grp BK21, San 31 Hyoja Dong/Pohang 790784//South Korea/ (REPRINT); Pohang Univ Sci & Technol, Dept Chem, Ctr Integrated Mol Syst, Funct Polymer Thin Film Grp BK21, Pohang 790784//South Korea/; Polymer Res Inst, Pohang 790784//South Korea/; Korea Res Inst Chem Technol, Adv Mat Div, Taejon 305606//South Korea/; Seoul Natl Univ, Sch Chem Engrg, Seoul 151742//South Korea/; Seoul Natl Univ, BK21 Program, Seoul 151742//South Korea/
 Journal: MACROMOLECULAR CHEMISTRY AND PHYSICS, 2002, V203, N5-6 (MAR 28), P 801-811
 ISSN: 1022-1352 Publication date: 20020328
 Publisher: WILEY-V C H VERLAG GMBH, PO BOX 10 11 61, D-69451 WEINHEIM, GERMANY

Language: English Document Type: ARTICLE

Abstract: Soluble poly(methylsilsequioxane) (PMSSQ) precursor is a potential dielectric candidate for advanced **microelectronic** devices based on a multilayer structure. Thermogravimetric and FT-IR spectroscopic analyses showed that the precursor undergoes a curing reaction between 100 and 450 degrees C. Time-resolved stress analysis of the PMSSQ films was used to measure the residual stress, which is critical to the reliability of films in the multilayer devices. The film stress fluctuates with temperature over the range 0-100 MPa during the curing process, but increases almost linearly with temperature during the subsequent cooling run. The final stress at room temperature ranged from 30 to 120 MPa, and was found to depend on factors such as the number of coatings, thickness, heating rate and steps, final cure temperature and degree of curing. The residual stress is caused by polymer chain immobilization and volume shrinkage due to the curing

reaction. This effect competes with the reduction in stress due to the relaxation of polymer chains by thermal heat. In particular the residual stress was found to induce cracks in films of thickness greater than 1000 nm during the cooling run after curing. In addition, the structure, refractive index, and dielectric constant of the cured films were determined using X-ray diffraction and ellipsometry. IT is recommended that PMSSQ films be processed at temperatures in the range 300-450degreesC and limited to thickness to less than 3000 nm.

24/3,AB/33 (Item 2 from file: 34)
 DIALOG(R)File 34:SciSearch(R) Cited Ref Sci
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09376946 Genuine Article#: 399UQ Number of References: 60
 Title: Laser-assisted metal deposition from liquid-phase precursors on polymers (ABSTRACT AVAILABLE)
 Author(s): Kordas K (REPRINT) ; Bekesi J; Vajtai R; Nanai L; Leppavuori S; Uusimaki A; Bali K; George TF; Galbacs G; Ignacz F; Moilanen P
 Corporate Source: Univ Oulu, Microelect & Mat Phys Labs, PL 4500/FIN-90570 Oulu//Finland/ (REPRINT); Univ Oulu, Microelect & Mat Phys Labs, FIN-90570 Oulu//Finland/; Univ Oulu, EMPART Res Grp Infotech Oulu, FIN-90570 Oulu//Finland/; Univ Szeged, Dept Expt Phys, H-6720 Szeged//Hungary/; Laser Lab Gottingen, D-37077 Gottingen//Germany/; Univ Szeged, Dept Phys, JGYTF, H-6720 Szeged//Hungary/; Univ Wisconsin, Dept Chem, Off Chancellor, Stevens Point//WI/54481; Univ Wisconsin, Dept Phys & Astron, Off Chancellor, Stevens Point//WI/54481; Univ Szeged, Dept Inorgan & Analyt Chem, H-6720 Szeged//Hungary/; Univ Szeged, Dept Opt & Quantum Elect, H-6720 Szeged//Hungary/
 Journal: APPLIED SURFACE SCIENCE, 2001, V172, N1-2 (MAR 1), P178-189
 ISSN: 0169-4332 Publication date: 20010301
 Publisher: ELSEVIER SCIENCE BV, PO BOX 211, 1000 AE AMSTERDAM, NETHERLANDS
 Language: English Document Type: ARTICLE
 Abstract: In this work, a short review is presented for results utilizing the technique of laser-assisted metallization of dielectrics. Experimental efforts and results related to the **metal** (palladium (Pd), **copper** (Cu) and silver (Ag)) deposition on **polymeric** materials (**polyimide** (PI), mylar) are reported. These polymers and metals are chosen due to their growing importance in the rapidly-developing microelectronics packaging industry. The method of laser-induced chemical liquid-phase deposition (LCLD) offers many advantages compared to other techniques, such as laser-induced forward transfer (LIFT), pulsed-laser deposition (PLD) and laser-assisted chemical vapor-phase deposition (LCVD). The LCLD is time and cost effective because vacuum tools and special pre-treatments are not required. The consumed chemicals used in precursors are non-harmful and easy to handle due to the liquid phase.

For the optimal physical and chemical properties of deposits, the laser and solution parameters are varied. XeCl and KrF excimer and Ar+ lasers are employed for executing the palladium. Ag and/or Cu formation on the polymer substrates. Chemical and physical analyses of the formed metal patterns are performed by EDX, XRD, FESEM, SEM, resistance and adhesion measurements. (C) 2001 Elsevier Science B.V. All rights reserved.

24/3,AB/34 (Item 3 from file: 34)
 DIALOG(R)File 34:SciSearch(R) Cited Ref Sci
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05897361 Genuine Article#: XF227 Number of References: 191
 Title: Multilevel interconnections for ULSI and GSI era (ABSTRACT
 AVAILABLE)
 Author(s): Murarka SP (REPRINT)
 Corporate Source: RENSSELAER POLYTECH INST,CTR ADV INTERCONNECT SCI &
 TECHNOL, CTR INTEGRATED ELECT & ELECT MFG/TROY//NY/12180 (REPRINT)
 Journal: MATERIALS SCIENCE & ENGINEERING R-REPORTS, 1997, V19, N3-4 (MAY 1)
 , P87-151
 ISSN: 0927-796X Publication date: 19970501
 Publisher: ELSEVIER SCIENCE SA LAUSANNE, PO BOX 564, 1001 LAUSANNE,
 SWITZERLAND

Language: English Document Type: REVIEW

Abstract: A high performance interconnection network on a **chip** is essential to match the ever improving performance of the semiconductor devices they interconnect. This issue reviews the need, some fundamental background justifying the need, approaches one can take in trying to satisfy the need, and associated issues related to the concept of multilevel interconnection (MLI) technology for the ULSI/GSI era of the silicon **integrated circuits**, with emphasis on materials and the processes that will lead to an acceptable MLI scheme. Thus besides discussing the MLI concepts and implementation issues, properties of metals and their alloys (especially those of Cu and Al), diffusion barrier/adhesion promoter, interlayer dielectrics, deposition and etching of materials, planarization issues, reliability issues, and new materials concepts are presented. Emphasis is obviously placed on the presently focused research on replacing aluminum with copper based interconnections. The author's viewpoint on gradual changes in replacing current materials with newer materials as they become available is also presented. A synergism between the materials sets for on-**chip** and off-**chip** interconnections has been pointed out.

24/3,AB/35 (Item 1 from file: 35)
 DIALOG(R)File 35:Dissertation Abs Online
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01784085 AADAAI9994397

Organic film and contaminant removal from surfaces in the manufacture of **integrated circuits**

Author: Chavez, Kristi Lynn (Albrecht)

Degree: Ph.D.

Year: 2000

Corporate Source/Institution: Georgia Institute of Technology (0078)

Source: VOLUME 61/11-B OF DISSERTATION ABSTRACTS INTERNATIONAL.

PAGE 5997. 127 PAGES

ISBN: 0-493-01243-5

This work focuses on the development of novel approaches to organic film removal, surface cleaning, and conditioning that ensures compatibility of this critical fabrication step with vacuum sequences in **integrated circuit** (IC) manufacturing. The general approach investigated for organic film and contaminant removal is the use of a fluid under elevated temperature and pressure conditions. A fluid in the vicinity of its boiling point remains a liquid by increasing the pressure; a reduction in pressure, however, results in liquid evaporation, permitting subsequent transfer of substrates into a vacuum chamber. An additional advantage of this approach is the elimination of a drying step.

Addition of carbon dioxide to water at 165°C and 850 psi enhances water interaction with organic materials, and thus provides a means to remove ion-implanted photoresist films up to an implant density of 10¹⁶ atoms/cm². Resist removal was also

demonstrated with this approach by use of acetic acid, although reasonable removal rates were obtained at much lower temperature and pressure conditions. Therefore, in order to slow the removal rates and thereby allow more detailed investigations of organic film removal by acetic acid, 35°C and atmospheric conditions were used. If an adhesion-promoting layer was applied to the silicon surface prior to the polymer application, a thin **polymeric** residue remained on the silicon surface.

Potential etching or roughening of the underlying surface was also studied. In the case of acetic acid, the native copper oxide inherent to air-exposed **copper films** was removed by acetic acid, leaving an oxide-free copper surface without attack of the underlying **metallic copper**.

As applied to a semiconductor manufacturing line, these approaches are capable of removing organic films from surfaces, yielding carbon concentrations comparable to those of an RCA-cleaned surface. Also, since a nitrogen dry is effective after the removal methods investigated, the need for a water-rinsing step essential to the RCA clean may be eliminated. Finally, this new cleaning approach minimizes the volume of solvents, acids, and de-ionized water used in IC photoresist removal, thereby greatly reducing the environmental impact of IC manufacture.

24/3,AB/36 (Item 2 from file: 35)
 DIALOG(R)File 35:Dissertation Abs Online
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01423319 AADAAI9521162

NUCLEATION AND GROWTH MECHANISMS OF **COPPER FILM** FROM MOCVD
 (THIN FILMS, CHEMICAL VAPOR DEPOSITION)

Author: KIM, JUNG-YEUL

Degree: PH.D.

Year: 1994

Corporate Source/Institution: UNIVERSITY OF KENTUCKY (0102)

Source: VOLUME 56/03-B OF DISSERTATION ABSTRACTS INTERNATIONAL.

PAGE 1649. 218 PAGES

Low pressure chemical vapor deposition of copper from copper(II) hexafluoroacetylacetonate Cu(hfa)_2 on different substrates has been investigated. The present work was undertaken to determine the effects of substrates on the nucleation and growth mechanisms of **copper CVD films** as a function of deposition temperature, precursor concentration, and water vapor concentration. Different initial film compositions and growth rates result in different types of substrates even at the same deposition conditions. No changes in surface morphology and electrical properties were observed for steady-state **copper films**. However, nucleation was strongly dependent upon the type of substrate used.

Minimal copper deposition was observed on the **polyimide** substrate in the absence of water vapor. The initial nucleation and growth of **copper film** on the edge of the **polyimide** substrate was initiated by the transport of reaction products from the **copper film** on the side wall of the silicon **wafer** rather than the surface reaction of the vapor phase precursor at the **polyimide** substrate surface. The presence of electron rich metallic substrates appears to be extremely important for the initial surface activation process between the vapor phase precursor and the substrate in the absence of water vapor according to the Langmuir-Hinshelwood type surface reaction schemes. When a 1500Å Au substrate was used, interdiffusion was observed in the **copper film** leading to the formation of Cu_3Au . Various alloy forms can be obtained as a function of deposition temperature.

When the optimum water vapor flow rate was used, an enhanced

deposition rates was obtained without sacrificing the **copper film** properties. The initially deposited films were in the form of copper oxide irrespective of the substrate used. The reduction of **copper oxide** to **metallic copper** can then proceed thermally or by a reaction with H_2 from either the processing gas or water vapor. When either low deposition temperatures or excess water vapor were employed, the reaction rate was too low for complete reduction to copper and a mixture of copper oxide and copper was formed in the deposited film. The results are used to develop an understanding of copper deposition mechanisms and the various film growth processes.

24/3,AB/37 (Item 3 from file: 35)
 DIALOG(R)File 35:Dissertation Abs Online
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01240002 AAD9227033
 INVESTIGATIONS OF NEW POTENTIOMETRIC GAS SENSING SYSTEMS (GAS SENSING, OXYGEN SENSORS)
 Author: YIM, HYOUNG-SIK
 Degree: PH.D.
 Year: 1992
 Corporate Source/Institution: THE UNIVERSITY OF MICHIGAN (0127)
 Source: VOLUME 53/05-B OF DISSERTATION ABSTRACTS INTERNATIONAL.
 PAGE 2288. 156 PAGES

Research concerning the development of new and/or improved potentiometric gas sensing systems is described. Studies relating to the development of reversible potentiometric oxygen sensors based on **polymeric** and metallic film electrodes are presented. In addition, the design and analytical utility of a novel differential ion-selective membrane electrode-based potentiometric gas sensing cell with enhanced sensitivity is documented.

The performance of a reversible potentiometric oxygen gas sensor based on a **polymeric** membrane doped with cobalt-complexes is described. For such sensors, the potentiometric oxygen response is attributed to a mixed potential originating from the underlying platinum electrode surface as well as the Co(II)-tetren doped film. This leads to a short term oxygen response of nearly the theoretical slope value of 118 mV/decade, below 10% O_2 . In the presence of the Co(II)-tetren/PVC film, an analytically useful response is observed for approx. 6-8 days.

Thin films of **metallic copper**, electrochemically deposited on platinum and sputtered on a single crystal silicon **wafer**, are also examined for reversible potentiometric oxygen sensing. The long-term reversibility and potentiometric stability of such **copper film**-based sensors is enhanced (up to one month) by preventing the formation of cuprous oxide on the surfaces via the application of an external non-polarizing cathodic current through the working electrode, or by specifically using sputtered **copper films** that have (100) crystal structures as determined by X-ray diffraction.

Finally, the development and application of a differential ion-selective membrane electrode-based potentiometric gas sensing cell is described. A novel design for devising static and automated flow-through type gas sensing systems with enhanced sensitivity involves the use of two working gas sensing electrode half-cells in a differential measurement arrangement. For the model analyte gas ammonia, response slopes approach the theoretically predicted value of 118 mV/decade. To demonstrate analytical utility, the flow-through arrangement is further used to determine ammonia-N concentrations in bioreactor media with good correlation with conventional electrode and enzymatic methods. The prospects of fabricating similar differential detection arrangements for

CO₂, NO₂, and SO₂ are also discussed.

24/3,AB/38 (Item 1 from file: 94)
 DIALOG(R)File 94:JICST-EPlus
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05007229 JICST ACCESSION NUMBER: 02A0020864 FILE SEGMENT: JICST-E
 Direct **Metallization** of **Copper** Using Surface Modification and
 Thermocompression of **Polyimide** Resin.
 IKEDA SHINGO (1); NAWAFUNE HIDEMI (1); MIZUMOTO SHOZO (1); AKAMATSU KENSUKE
 (2); SEITA MASARU (3)
 (1) Konandai Daigakuinshizenkagakukenkkyuka; (2) Konandai Riko; (3) LeaRonai
 Japan Inc.
 Erektoronikusu Jisso Gakkaishi(Journal of Japan Institute of Electronics
 Packaging), 2001, VOL.4,NO.7, PAGE.603-606, FIG.9, TBL.1, REF.4
 JOURNAL NUMBER: S0579CAN ISSN NO: 1343-9677
 UNIVERSAL DECIMAL CLASSIFICATION: 621.3.049.75 621.793.3+621.357.7
 LANGUAGE: Japanese COUNTRY OF PUBLICATION: Japan
 DOCUMENT TYPE: Journal
 ARTICLE TYPE: Original paper
 MEDIA TYPE: Printed Publication

ABSTRACT: A novel method is presented for the direct **metallization** of
copper on a semi-cured **polyimide** resin substrates by the
 process of carboxylation of the resin surface, adsorption and reduction
 of coppier(II) ions followed by thermocompression bonding. The
 potassium hydroxide treatment on **polyimide** resin lead to cleavage
 of imide ring to form the carboxyl group onto the resin surface, being
 responsible for the adsorption of copper(II) ions. The conductivity of
 the **copper** thin film formed by the reduction process was
 over $1.6 \times 10^{-2} \text{ S/}\cdot\text{SQU}$. which was suitable for subsequent copper
 electroplating. The peel strength between **polyimide** film
 and **copper** film was reached to be 1.10kN/m by
 thermocompression at 300.DEG.C. under a pressure of 24.5MPa. (author
 abst.)

24/3,AB/39 (Item 2 from file: 94)
 DIALOG(R)File 94:JICST-EPlus
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03401174 JICST ACCESSION NUMBER: 97A0898499 FILE SEGMENT: JICST-E
 Metallization on **Polyimide** Film by Ion and Vapor Deposition (IVD)
 Method.
 EBE A (1); SETSUHARA Y (1); MIYAKE S (1)
 (1) Osaka Univ., Osaka, JPN
 Trans JWRI(Weld Res Inst Osaka Univ), 1996, VOL.25,NO.1, PAGE.25-30, FIG.8,
 REF.9
 JOURNAL NUMBER: S0875AAW ISSN NO: 0387-4508
 UNIVERSAL DECIMAL CLASSIFICATION: 621.315.5 678.029.6/.8
 LANGUAGE: English COUNTRY OF PUBLICATION: Japan
 DOCUMENT TYPE: Journal
 ARTICLE TYPE: Original paper
 MEDIA TYPE: Printed Publication

ABSTRACT: A study was performed to investigate the effect of argon ion
 bombardment for improvement of **copper** film adhesion on
polyimide film substrates. The thin **copper** films were
 prepared on **polyimide** films by evaporation of **copper**
metal and simultaneous irradiation by argon ions with energies in
 the range of 0.5 to 1 0.0keV. The argon ion density irradiated at the
 interface between the **copper** thin film and the substrate

was changed from 5×10^{14} to 5×10^{16} ions/cm² in each ion energy range. The adhesion of **copper films** was evaluated by means of peel strength. The **copper films** prepared with 5.0 keV argon ions have strong adhesion, but the adhesion of **copper films** with 5.0 keV and 10.0 keV argon ions was lower than that of **copper films** prepared without argon ion bombardment. The chemical states of the **polyimide** film surface and the chemical binding states at the interlayer were evaluated by X-ray photoelectron spectrometry. The structure of the interface between the **copper film** and the **polyimide** substrate was analyzed by transmission electron microscopy. The argon ion bombardment carbonized the film surface, and the increase of the carbonization caused the decrease of the adhesion. The increase of the adhesion did not depend on the change of chemical binding states at the interlayer. The interface structures prepared by ion bombardment have a mixed layer consisting of the copper atoms diffused into the substrate and the carbon atoms from the **polyimide**. The formation of the mixed layer by ion bombardment contributed to improve the **copper film** adhesion. (author abst.)

24/3,AB/40 (Item 3 from file: 94)
 DIALOG(R)File 94:JICST-EPlus
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01928599 JICST ACCESSION NUMBER: 93A1001934 FILE SEGMENT: JICST-E
 Metallization on Polymer by Ion and Vapor Deposition.
 EBE AKINORI (1); IMAI OSAMU (1); OGATA KIYOSHI (1); KUBOSHIMA RYUICHIRO (1); DOI AKIRA (1); SUZUKI YASUO (1)
 (1) Nissin Electric Co., Ltd.
 Nisshin Denki Giho(Nissin Electric Review), 1993, VOL.38,NO.2, PAGE.18-24, FIG.10, TBL.1, REF.3
 JOURNAL NUMBER: S0817BAJ ISSN NO: 0549-5377 CODEN: NIDGA
 UNIVERSAL DECIMAL CLASSIFICATION: 621.382.002.2 539.23:669
 LANGUAGE: Japanese COUNTRY OF PUBLICATION: Japan
 DOCUMENT TYPE: Journal
 ARTICLE TYPE: Original paper
 MEDIA TYPE: Printed Publication
 ABSTRACT: Adhesion of **copper films** on **polyimide** substrates is becoming important for **microelectronic** applications, such as multilayer packaging and flexible circuit boards. However, the adhesion was not strong for these applications, and the technology for **copper metallization** on the **polyimide** with strong adhesion has been studied by many researchers. In this paper, the **copper films** were prepared on **polyimide** film substrates by evaporation of **copper metal** and simultaneous bombardment argon ion with 0.5-10keV (IVD method). The adhesion of the samples was measured by using of a 90.DEG. peel test method. The surface chemistry of the substrates and chemical binding states at the interlayer were evaluated by Fourier transform infrared spectroscopy and X-ray photoelectron spectrometry. The effect of argon ion bombardment on the adhesion was discussed. (author abst.)

24/3,AB/41 (Item 1 from file: 144)
 DIALOG(R)File 144:Pascal
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14535662 PASCAL No.: 00-0200413
 The effect of **polyimide** passivation on the electromigration in sputtered **copper films**

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Journal: The International journal of microcircuits and electronic
packaging, 1999, 22 (4) 395-400

Language: English

Electromigration damage (EMD) is one of the major causes for the failures of interconnect. In this study, the electromigration of Cu with **polyimide** passivation is investigated with an isothermal resistance change method. Resistance decreases in passivated samples caused by diffusion of **polyimide** ingredients into copper are observed. **Polyimide** passivation enhances the lifetime of the Cu metallization (sputtered **copper films**). The lifetime of passivated **copper films** are from 1.1 to 29 times those of the unpassivated ones, depending on the passivation materials and the current stressing conditions. Rigidity of the passivation is found to be the controlling factor for lifetime improvement. In addition, the adhesion strength between the Cu and the **polyimide** is studied in this work. An interlayer TiWN promotes the adhesion between the Cu and the **polyimide** interface.

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24/3,AB/42 (Item 2 from file: 144)

DIALOG(R)File 144:Pascal

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13781856 PASCAL No.: 98-0495597

Electromigration study of Al/low dielectric constant polymer and Al/SiO
SUB 2 dioxide line structures

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Texas 78741

Journal: Journal of applied physics, 1998-12-01, 84 (11) 6007-6016

Language: English

Electromigration reliability is a key concern for implementation of low dielectric constant (low kappa) materials for on-chip interconnects. To address this problem, we have carried out a comparative study of electromigration performance for Al(0.5 wt% Cu) line structures formed with tetraethylorthosilicate (TEOS)-based SiO SUB 2 and a low kappa **polymeric** dielectric, poly(arylene ether) (PAE). The test structures consisted of 800 mu m long single-level line structures with adjacent extrusion monitor lines. Resistometric measurements were performed to measure electromigration lifetime. To supplement lifetime measurements, x-ray diffraction and transmission electron microscopy were used to measure overall grain size and grain orientation distributions, and electron backscattering diffraction was applied to determine local grain orientation and grain boundary characteristics. In addition, scanning electron microscopy was used to examine void morphology. Microstructural analysis has revealed a larger grain size and enhanced <left angle bracket>111<right angle bracket> texture in the PAE passivated Al lines compared to TEOS SiO SUB 2 passivated lines. The difference in microstructure is attributed to a high temperature process used to cure the PAE which is absent from the processing of the TEOS SiO SUB 2 samples. The improved electromigration performance can be attributed to the larger grain size and decreased grain misorientation in the PAE passivated lines. (c) 1998 American Institute of Physics.

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24/3,AB/43 (Item 3 from file: 144)
 DIALOG(R)File 144:Pascal
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13209360 PASCAL No.: 97-0476033
 Laser induced deposition of tungsten and copper
 MEUNIER M; IZQUIERDO R; TABBAL M; EVOY S; DESJARDINS P; BERNIER M H;
 BERTOMEU J; ELYAAGoubi N; SUYS M; SACHER E; YELON A
 Groupe des Couches Minces and Departement de Genie Physique, École
 Polytechnique de Montreal, Quebec, Canada
 Journal: Materials science & engineering. B, Solid-state materials for
 advanced technology, 1997, 45 (1-3) 200-207
 Language: English Summary Language: English
 Copyright (c) 1997 Elsevier Science S.A. All rights reserved. We present
 recent results obtained in our laboratory on the laser processing for
 deposition of tungsten and copper. Four processes are presented. (1) An Ar
 SUP + laser or a diode laser are used in a direct writing mode to produce W
 and WSi SUB x lines from WF SUB 6 reduced by H SUB 2 and/or SiH SUB 4 , and
 deposited on silicon, TiN, SiO SUB x N SUB y and **polyimide**. (2) Pure
 &agr;-W films were also deposited on GaAs by KrF excimer laser-induced
 deposition using a WF SUB 6 /SiH SUB 4 /H SUB 2 gas mixture. (3) Very
 uniform, shiny **metallic copper films**, with Cu/C
 ratios up to 17 were deposited on TiN and fluoropolymer substrates by the
 KrF excimer laser-assisted decomposition of Cu(hfac)(TMVS). (4) Finally, we
 have studied the Ar SUP + and diode laser direct writing of Pd and Au for
 the selective activation of the electroless plating of Cu.

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24/3,AB/44 (Item 4 from file: 144)
 DIALOG(R)File 144:Pascal
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13123220 PASCAL No.: 97-0105366
 Gap fill dependence of fluorinated **polyimide** films on solid
 content, adhesion promoter, spin dwell time, and solvent spray
 WANG S; ZHAO B
 Sematech, Austin, Texas 78 741
 Journal: Journal of vacuum science & technology. B. Microelectronics and
 nanometer structures. Processing, measurement and phenomena, 1996-07, 14 (4)
 2656-2659
 Language: English
 Cross sectional scanning electron microscopy was used to study the
 dependence of gap fill capabilities in 0.25-2.0 μ m wide Al-Cu
metal gaps of DuPont fluorinated **polyimide** films on solid
 content, adhesion promoter, spin dwell time, and solvent spray. It was
 found that reduced solid content, application of adhesion promoter and
 solvent spray improve gap fill capabilities. However, these improvements
 are insignificant. Spin dwell time has little effect on gap fill. (c) 1996
 American Vacuum Society

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24/3,AB/45 (Item 5 from file: 144)
 DIALOG(R)File 144:Pascal
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13105163 PASCAL No.: 97-0087307

Characteristics of gap fill and planarization of fluorinated
polyimide FPI-45M films

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Journal: Journal of vacuum science & technology. B. Microelectronics and
nanometer structures. Processing, measurement and phenomena, 1996-01, 14 (1)
1) 174-178

Language: English

Cross sectional scanning electron microscopy was used to characterize the
gap fill and planarization properties of DuPont experimental fluorinated
polyimide FPI-45M films in Al-Cu metal gaps (0.25
to 2.0 μm in width) and over Al-Cu metal lines/spaces
(0.50/0.50 to 50/100 μm in lines/spaces). It was found that the gap fill
and planarization properties were dependent on the orientation of the
lengthwise direction of the Al-Cu metal gaps and lines/spaces
relative to the radius. Slightly enhanced fill is observed when gaps are
parallel to the local radius. Planarization over small lines/spaces is
identical regardless of their orientations. However, for large
lines/spaces, planarization values are significantly higher when the
line/space orientation is 90 Degree relative to the radius than those
oriented 0 Degree relative to the radius. (c) 1996 American Vacuum Society

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24/3,AB/46 (Item 1 from file: 103)

DIALOG(R)File 103:Energy SciTec

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04927204 SNL

Title: LDRD 10729 Ultra Miniaturization of RF using Microwave Chip on
Flex Technology, FY02 Final Report

Author(s): SANDOVAL, CHARLIE E.; WOUTERS, GREGG A.; SLOAN, GEORGE R.
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Corporate Source: Sandia National Labs., Albuquerque, NM (US); Sandia
National Labs., Livermore, CA (US)

Sponsoring Organization: US Department of Energy (US)

Source: PBD: 1 Mar 2003 Scope: FINAL

Publication Date: 20030301

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Report Number(s): SAND2003-0750

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Contract Number (Non-DOE): TRN US200307%%740

Language: English

Medium/Dimensions: 41 pages ; PDFN

Abstract: This report describes the activities on the ''Ultra
Miniaturization of RF'' project conducted as part of Sandia's
Laboratory Directed Research and Development (LDRD) program. The
objective was to evaluate a multichip module technology known as
Microwave Chip on Flex (MCOF)[1], which is a newer form of the
standard high density interconnect (HDI) technology originally
developed by General Electric and Lockheed Martin[2,3]. The program was
a three-year effort. In the first year, the team focused on
understanding the technology and developing a basic design library. In
the second year, devices and interconnects used at L, X, and Ku
frequency bands were evaluated via a test coupon (with no application
specific circuit design). In the third year, we designed, fabricated,
and evaluated a specific Ku-band circuit. The circuit design and layout
was performed by Sandia, and the module fabrication was performed by

Lockheed Martin Government Electronic Systems. In MCOF technology[1], bare **die** are placed face down on an adhesive backed flex circuit. The first level of the circuit is a pre-patterned titanium **copper** thin **film metal** system on a **polyimide** dielectric material. The complete module is then framed and filled with an epoxy encapsulant. The module is flipped and via holes are laser drilled through subsequent interconnect layers. Each addition layer is adhered to the top of the module and laser drilling repeated. The baseline design consisted of the original pre-patterned layer plus two additional metal layers. The base of the module is then machined so the heat spreader and frame are planar for a good thermal and electrical connection to the next assembly. This report describes the efforts conducted to evaluate the technology and its applicability to Sandia RF systems.

29/3,AB/1 (Item 1 from file: 2)
 DIALOG(R)File 2:INSPEC
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7103070 INSPEC Abstract Number: A2002-01-8115L-031, B2002-01-0520J-019
 Title: Analysis of current-potential hysteresis during electrodeposition of copper with additives
 Author(s): Hebert, K.R.
 Author Affiliation: Dept. of Chem. Eng., Iowa State Univ., Ames, IA, USA
 Journal: Journal of the Electrochemical Society vol.148, no.11 p. C726-32
 Publisher: Electrochem. Soc,
 Publication Date: Nov. 2001 Country of Publication: USA
 CODEN: JESOAN ISSN: 0013-4651
 SICI: 0013-4651(200111)148:11L:C726:ACPH;1-Z
 Material Identity Number: J010-2001-013
 U.S. Copyright Clearance Center Code: 0013-4651/2001/148(11)/726/7/\$7.00
 Language: English

Abstract: Two models are described for cyclic voltammetry during electrodeposition of copper from an acid sulfate bath containing the additives polyethylene glycol (PEG), chloride ions, and mercapto-1-propanesulfonate (MPSA). The same bath may be used for superfilling of cavities during fabrication of **copper on-chip metallization**. Experimental current-potential scans show a characteristic hysteresis in solutions with all three additives, which demonstrates the presence of both activated and inhibited kinetics. Both models assume that deposition is inhibited by a PEG-chloride adsorbed surface complex, as established previously. One model further hypothesizes that PEG is incorporated into grain boundaries in the copper at the same fractional coverage found on the external surface. The second model neglects PEG incorporation, but assumes that adsorbing MPSA directly displaces PEG from the surface. Both models predict hysteresis quantitatively similar to experiments, without the use of fitting parameters. The competitive adsorption model is favored because, unlike the PEG incorporation model, it yields realistic predictions of carbon and sulfur concentrations in the deposit.

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29/3,AB/2 (Item 2 from file: 2)
 DIALOG(R)File 2:INSPEC
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7052751 INSPEC Abstract Number: B2001-11-0170G-003
 Title: Ethylene glycol ether free solder paste development
 Author(s): Lytle, B.; Fang, T.; Li Li; Zhang, C.
 Author Affiliation: Motorola Inc., Tempe, AZ, USA
 Journal: Journal of Electronic Materials vol.30, no.8 p.1035-41
 Publisher: TMS; IEEE,
 Publication Date: Aug. 2001 Country of Publication: USA
 CODEN: JECMA5 ISSN: 0361-5235
 SICI: 0361-5235(200108)30:8L:1035:EGEF;1-S
 Material Identity Number: J246-2001-010
 U.S. Copyright Clearance Center Code: 0361-5235/2001/\$7.00
 Language: English

Abstract: There are growing concerns in the electronics industry for not only finding alternatives to lead but also other potentially hazardous materials as well. This paper summarizes the development of ethylene glycol ether (EGE)-free solder flux for the formulation of lead-free

solder pastes. Replacing the toxic components in the flux was only the first challenge, the criteria of commercially proven pastes also had to be met. Both commercial and in-house solder paste formulations were evaluated for printability, reflow, wetting, flux residue removal, and solder void characteristics. Two critical issues, solder bump voids and flux residue removal, were identified and associated with the high temperature reflow of Pb-free pastes. These issues were not effectively improved by the existing commercial EGE-free solder pastes. New solder paste formulations were developed utilizing alternative chemistry than those found in traditional solder paste fluxes. These pastes, some of which are also water soluble, reduced void frequency and size by more than 4* as compared to vendors pastes. Solder bump height uniformity of 135+or-4 μ m within each die was consistently achieved. Thermal-mechanical reliability tests were performed on various lead-free solder alloys using the new flux formulations. The reliability of flip chip assembled DCA on organic boards with both OSP/Cu and Cu/Ni/Au pad metallizations were comparable to eutectic Sn63Pb37 bumped assemblies using commercial pastes.

Subfile: B

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29/3,AB/3 (Item 3 from file: 2)

DIALOG(R)File 2:INSPEC

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6783438 INSPEC Abstract Number: B2001-01-2550F-029

Title: Application of an electrochemical **copper metallization** -planarization process to sub-0.25 μ m features

Author(s): Sun, J.J.; Taylor, E.J.; Inman, M.E.; Leedy, K.D.; Cortez, R.

Author Affiliation: Faraday Technol. Inc., Clayton, OH, USA

Conference Title: Interconnect and Contact Metallization for ULSI. Proceedings of the International Symposium (Electrochemical Society Proceedings Vol.99-31) p.152-61

Editor(s): Mathad, G.S.; Rathore, H.S.; Arita, Y.

Publisher: Electrochem. Soc, Pennington, NJ, USA

Publication Date: 2000 Country of Publication: USA ix+340 pp.

ISBN: 1 56677 254 0 Material Identity Number: XX-2000-01537

Conference Title: Interconnect and Contact Metallization for ULSI. Proceedings of the International Symposium

Conference Sponsor: Electrochem. Soc

Conference Date: 17-22 Oct. 1999 Conference Location: Honolulu, HI, USA

Language: English

Abstract: This paper describes an innovative charge modulated electrochemical deposition (CM-ECD) process to metallize and planarize semiconductor features. The process uses a high frequency charge modulated waveform to control the current distribution, and therefore the distribution of copper in trenches. By properly tuning the CM-ECD process parameters, semiconductor features are metallized without the need for "super-filling" brighteners, levelers, and/or suppressors. In addition, the CM-ECD process is conducted in the presence of polyethylene glycol (PEG)/chloride additives to promote copper recrystallization. Finally, data is presented demonstrating the ability to produce void and defect free copper filled trenches in the size range of 0.25 μ m to 8.0 μ m in the same, easily controlled copper sulfate/sulfuric acid-PEG/chloride plating bath.

Subfile: B

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29/3,AB/4 (Item 4 from file: 2)

DIALOG(R)File 2:INSPEC

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6659358 INSPEC Abstract Number: B2000-09-2550F-018

Title: Fundamental **study** on electroless **copper** plating bath applied for ULSI wiring

Author(s): Matsuoka, M.; Odani, M.; Taniwaki, M.; Kamo, Y.; Tamaki, J.; Yamamoto, Y.; Izumi, N.; Kamisawa, A.

Author Affiliation: Dept. of Chem., Ritsumeikan Univ., Kyoto, Japan

Journal: Memoirs of the Institute of Science & Engineering, Ritsumeikan University no.58 p.35-41

Publisher: Ritsumeikan Univ,

Publication Date: 1999 Country of Publication: Japan

CODEN: RDRKAJ ISSN: 0370-4254

SICI: 0370-4254(1999)58L:35:FSEC;1-I

Material Identity Number: F220-2000-001

Language: English

Abstract: As a novel wiring method, electroless **copper metallization** attracts intense attention to produce high reliability and high density interconnection of submicron features. Electroless copper plating has high potential for uniform filling of complicated trenches and vias. These features of electroless plating are excellent as compared with electroplating and CVD. In practical use of electroless copper plating, adhesion, resistivity, internal stress and crystal structure of the deposit are important factors. In this study, the effect of chemical composition of electroless copper solution and operating conditions on the properties of the deposits was systematically investigated. As the selective deposition of copper on barrier surface such as TiN is another key technology in fabricating ULSI circuit, the selectivity of copper deposition is also examined at different dissolved oxygen levels. Internal stress was reduced by using low-pH plating solution and 2,2'-bipyridyl. Addition of polyethylene glycol functioning as a surfactant gave a favorable effect on coating property unless it was codeposited in the film. The selective seeding of Pd and Ag was achieved by using a solution based on nitric acid. Fine circuit patterns on silicon **wafer** were metallized uniformly by reducing dissolved oxygen level in electroless copper solution.

Subfile: B

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29/3,AB/5 (Item 5 from file: 2)

DIALOG(R)File 2:INSPEC

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6599846 INSPEC Abstract Number: B2000-07-2550F-003

Title: Copper electroplating for future ultralarge scale integration interconnection

Author(s): Gau, W.C.; Chang, T.C.; Lin, Y.S.; Hu, J.C.; Chen, L.J.; Chang, C.Y.; Cheng, C.L.

Author Affiliation: Dept. of Chem. Eng., Chung-Yuan Univ., Chung Li, Taiwan

Journal: Journal of Vacuum Science & Technology A (Vacuum, Surfaces, and Films) Conference Title: J. Vac. Sci. Technol. A, Vac. Surf. Films (USA) vol.18, no.2 p.656-60

Publisher: AIP for American Vacuum Soc,

Publication Date: March 2000 Country of Publication: USA

CODEN: JVTAD6 ISSN: 0734-2101

SICI: 0734-2101(200003)18:2L:656:CEFU;1-E

Material Identity Number: D746-2000-003

U.S. Copyright Clearance Center Code: 0734-2101/2000/18(2)/656(5)/\$15.00

Conference Title: Proceedings of 9th Canadian Semiconductor Technology Conference

Conference Date: 10-13 Aug. 1999 Conference Location: Ottawa, Ont., Canada

Language: English

Abstract: Copper has received considerable attention during the past few years because of its low electrical resistivity, high melting temperature, and high electromigration resistance. Since electrochemical plating has several advantages compared to physical vapor deposition and chemical vapor deposition due to its low cost and low processing temperature, it becomes the most attractive technique for the implementation of **Cu metallization**. In this article, we report an electroplating scheme for **Cu** deposition and **study** aspects of **copper** electroplating related to chemical additives effected on Cu deposition. A low resistivity Cu film ($\sim 2 \mu\Omega \text{ cm}$) could be obtained when metallic ion concentration is decreased or current density is increased. The detailed correlation between film resistivity, electrolyte concentration, current density, and film morphology is given in this study. In addition, chemical additives in electrolyte solutions also play important roles in copper electroplating. In this report, thiourea and polyethylene glycol, which are usually added in printing circuit board electroplating to influence nucleation, have been used as gap filling promoters to help Cu filling. The results show that addition of thiourea and polyethylene glycol could help in forming smooth Cu film but does not promote Cu filling ability. Hydroxyl amine sulfate, which has both amino and sulfate groups, is proposed for use as a gap filling promoter in helping Cu electroplating. In this work, we demonstrate that Cu could be electroplated into fine trenches (at $0.3 \mu\text{m}$ dimension with aspect ratio of 3) when hydroxyl amine sulfate is present and no voids are formed.

Subfile: B

Copyright 2000, IEE

29/3,AB/6 (Item 6 from file: 2)

DIALOG(R)File 2:INSPEC

(c) 2003 Institution of Electrical Engineers. All rts. reserv.

5869050 INSPEC Abstract Number: A9809-6475-004, B9805-0560-004

Title: The dissolution of copper in common solvents used for low dielectric polymers

Author(s): Purdy, A.P.; Godbey, D.; Buckley, L.

Author Affiliation: Div. of Chem., Naval Res. Lab., Washington, DC, USA

Journal: Thin Solid Films Conference Title: Thin Solid Films (Switzerland) vol.308-309 p.486-9

Publisher: Elsevier,

Publication Date: 31 Oct. 1997 Country of Publication: Switzerland

CODEN: THSFAP ISSN: 0040-6090

SICI: 0040-6090(19971031)308/309L.486:DCCS;1-P

Material Identity Number: T070-98002

U.S. Copyright Clearance Center Code: 0040-6090/97/\$17.00

Conference Title: 24th International Conference on Metallurgical Coatings and Thin Films

Conference Date: 21-25 April 1997 Conference Location: San Diego, CA, USA

Language: English

Abstract: A major issue with **copper metallization** is the diffusion and/or dissolution of copper into polymers used as insulating dielectric layers in **microelectronic** devices. Polymers are typically deposited by spin-coating. But when substrate-polymer interactions are studied, the process by which the polymer is deposited must be carefully considered. For example, what roles do the solvent or the curing conditions

play in bringing copper into the polymer film? In this study, we focus on a frequently neglected aspect of the problem - that of solvent interactions with the copper. We have examined the ability of commonly used solvents to dissolve copper under oxygen atmospheres. For this **study**, **copper** turnings were stirred with the pure solvent under O/sub 2/. The presence of copper in solution was determined with spot tests, and solid reaction products were characterized with X-ray powder diffraction. Copper was found to dissolve in 1-methyl-2-pyrrolidinone, anisole, methylisobutylketone, propylene **glycol** methyl ether acetate, gamma-butyrolactone, dimethylacetamide, water, methylmethacrylate, m-cresol, and sulfolane in the presence of oxygen, but not in toluene, chlorobenzene, dichlorobenzene, chloroform, acetone and tetrahydrofuran.

Subfile: A B

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29/3,AB/7 (Item 7 from file: 2)

DIALOG(R)File 2:INSPEC

(c) 2003 Institution of Electrical Engineers. All rts. reserv.

02212686 INSPEC Abstract Number: A84036029

Title: The determination of acidic degradation products in aqueous ethylene **glycol** and propylene **glycol** solutions using ion chromatography

Author(s): Rossiter, W.J., Jr.; Brown, P.; Godette, M.

Author Affiliation: Center for Building Technol., US Dept. of Commerce, Washington, DC, USA

Journal: Solar Energy Materials vol.9, no.3 p.267-79

Publication Date: Oct.-Dec. 1983 Country of Publication: Netherlands

CODEN: SOEMDH ISSN: 0165-1633

U.S. Copyright Clearance Center Code: 0165-1633/83/0000-0000/\$03.00

Language: English

Abstract: Aqueous ethylene **glycol** and propylene **glycol** solutions are the principal antifreezes used as heat transfer fluids in flat-plate solar collector systems. Acidic degradation products formed in the thermal oxidation of these solutions have been analyzed using ion chromatography (IC) and the ion-chromatography exclusion (ICE) method of analysis. The primary acidic degradation product found in the case of ethylene **glycol** solutions was glycolic acid. Propylene **glycol** solutions yielded mainly lactic acid. The presence of **metallic copper** significantly increased the amount of degradation products over that produced in the absence of metal. Solutions heated in the presence of metallic aluminum were only slightly more degraded than those heated without metal, but contained relatively large amounts of formic acid. Only traces of formic acid were observed when the **glycol** solutions were heated in the absence of metal or in the presence of **metallic copper**.

Subfile: A

29/3,AB/8 (Item 1 from file: 8)

DIALOG(R)File 8:Ei Compendex(R)

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05934635

E.I. No: EIP01456719841

Title: Ethylene **glycol** ether free solder paste development

Author: Lytle, B.; Fang, T.; Li, L.; Zhang, C.

Corporate Source: Motorola Semiconductor Products Sector, Tempe, AZ 85284, United States

Source: Journal of Electronic Materials v 30 n 8 August 2001. p 1035-1041

Publication Year: 2001

CODEN: JECMA5 ISSN: 0361-5235

Language: English

Abstract: There are growing concerns in the electronics industry for not only finding alternatives to lead but also other potentially hazardous materials as well. This paper summarizes the development of ethylene glycol ether (EGE)-free solder flux for the formulation of lead-free solder pastes. Replacing the toxic components in the flux was only the first challenge, the criteria of commercially proven pastes also had to be met. Both commercial and in-house solder paste formulations were evaluated for printability, reflow, wetting, flux residue removal, and solder void characteristics. Two critical issues, solder bump voids and flux residue removal, were identified and associated with the high temperature reflow of Pb-free pastes. These issues were not effectively improved by the existing commercial EGE-free solder pastes. New solder paste formulations were developed utilizing alternative chemistry than those found in traditional solder paste fluxes. These pastes, some of which are also water soluble, reduced void frequency and size by more than 4x as compared to vendors' pastes. Solder bump height uniformity of 135 plus or minus 4 μ m within each die was consistently achieved. Thermal-mechanical reliability tests were performed on various lead-free solder alloys using the new flux formulations. The reliability of flip chip assembled DCA on organic boards with both OSP/Cu and Cu/Ni/Au pad metallizations were comparable to eutectic Sn63Pb37 bumped assemblies using commercial pastes. 8 Refs.

29/3,AB/9 (Item 2 from file: 8)

DIALOG(R)File 8: Ei Compendex(R)

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04937567

E.I. No: EIP98024056760

Title: Dissolution of copper in common solvents used for low dielectric polymers

Author: Purdy, Andrew P.; Godbey, David; Buckley, Leonard

Corporate Source: Naval Research Lab, Washington, DC, USA

Conference Title: Proceedings of the 1997 24th International Conference on Metallurgical Coatings and Thin Films

Conference Location: San Diego, CA, USA Conference Date: 19970421-19970425

E.I. Conference No.: 47640

Source: Thin Solid Films v 308-309 Oct 31 1997. p 486-489

Publication Year: 1997

CODEN: THSFAP ISSN: 0040-6090

Language: English

Abstract: A major issue with copper metallization is the diffusion and/or dissolution of copper into polymers used as insulating dielectric layers in microelectronic devices. Polymers are typically deposited by spin-coating. But when substrate-polymer interactions are studied, the process by which the polymer is deposited must be carefully considered. For example, what roles do the solvent or the curing conditions play in bringing copper into the polymer film? In this study, we focus on a frequently neglected aspect of the problem - that of solvent interactions with the copper. We have examined the ability of commonly used solvents to dissolve copper under oxygen atmospheres. For this study, copper turnings were stirred with the pure solvent under O₂. The presence of copper in solution was determined with spot tests, and solid reaction products were characterized with X-ray powder diffraction. Copper was found to dissolve in 1-methyl-2-pyrrolidinone, anisole, methylisobutylketone, propylene glycol methyl ether acetate, gamma

-butyrolactone, dimethylacetamide, water, methylmethacrylate, m-cresol, and sulfolane in the presence of oxygen, but not in toluene, chlorobenzene, o-dichlorobenzene, chloroform, acetone, ethylacetate and tetrahydrofuran. (Author abstract) 9 Refs.

29/3,AB/10 (Item 3 from file: 8)
 DIALOG(R)File 8:EI Compendex(R)
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01511077

E.I. Monthly No: EI8404037380
 E.I. Yearly No: EI84119420

Title: DETERMINATION OF ACIDIC DEGRADATION PRODUCTS IN AQUEOUS ETHYLENE GLYCOL AND PROPYLENE GLYCOL SOLUTIONS USING ION CHROMATOGRAPHY.

Author: Rossiter, Walter J. Jr.; Brown, Paul W.; Godette, McClure
 Corporate Source: NBS, Building Materials Div, Washington, DC, USA
 Source: Solar Energy Materials v 9 n 3 Oct-Dec 1983 p 267-279
 Publication Year: 1983

CODEN: SOEMDH ISSN: 0165-1633

Language: ENGLISH

Abstract: Aqueous ethylene **glycol** and propylene **glycol** solutions are the principal antifreezes used as heat transfer fluids in flat-plate solar collector systems. Acidic degradation products formed in the thermal oxidation of these solutions have been analyzed by using ion chromatography (IC) and the ion-chromatography exclusion (ICE) method of analysis. The primary acidic degradation product found in the case of ethylene **glycol** solutions was glycolic acid. Propylene **glycol** solutions yielded mainly lactic acid. The presence of **metallic copper** significantly increased the amount of degradation products over that produced in the absence of metal. Solutions heated in the presence of metallic aluminum were only slightly more degraded than those heated without metal, but contained relatively large amounts of formic acid. 18 refs.

29/3,AB/11 (Item 1 from file: 34)
 DIALOG(R)File 34:SciSearch(R) Cited Ref Sci
 (c) 2003 Inst for Sci Info. All rts. reserv.

10092996 Genuine Article#: 483TZ Number of References: 40
 Title: Analysis of current-potential hysteresis during electrodeposition of copper with additives (ABSTRACT AVAILABLE)

Author(s): Hebert KR (REPRINT)
 Corporate Source: Iowa State Univ, Dept Chem Engr, Ames//IA/50011 (REPRINT);
 Iowa State Univ, Dept Chem Engr, Ames//IA/50011
 Journal: JOURNAL OF THE ELECTROCHEMICAL SOCIETY, 2001, V148, N11 (NOV), P C726-C732

ISSN: 0013-4651 Publication date: 20011100

Publisher: ELECTROCHEMICAL SOC INC, 65 SOUTH MAIN STREET, PENNINGTON, NJ 08534 USA

Language: English Document Type: ARTICLE

Abstract: Two models are described for cyclic voltammetry during electrodeposition of copper from an acid sulfate bath containing the additives polyethylene **glycol** (PEG), chloride ions, and mercapto-1-propanesulfonate (MPSA). The same bath may be used for superfilling of cavities during fabrication of **copper on-chip metallization**. Experimental current-potential scans show a characteristic hysteresis in solutions with all three additives, which demonstrates the presence of both activated and inhibited kinetics. Both models assume that deposition is inhibited by a

PEG-chloride adsorbed surface complex, as established previously. One model further hypothesizes that PEG is incorporated into grain boundaries in the copper at the same fractional coverage found on the external surface. The second model neglects PEG incorporation, but assumes that adsorbing MPSA directly displaces PEG from the surface. Both models predict hysteresis quantitatively similar to experiments, without the use of fitting parameters. The competitive adsorption model is favored because, unlike the PEG incorporation model, it yields realistic predictions of carbon and sulfur concentrations in the deposit. (C) 2001 The Electrochemical Society.

29/3,AB/12 (Item 2 from file: 34)
 DIALOG(R)File 34:SciSearch(R) Cited Ref Sci
 (c) 2003 Inst for Sci Info. All rts. reserv.

09907400 Genuine Article#: 463CD Number of References: 7
 Title: Ethylene **glycol** ether free solder paste development (ABSTRACT AVAILABLE)
 Author(s): Lytle B (REPRINT) ; Fang T; Li L; Zhang C
 Corporate Source: Motorola Inc, Semicond Prod Sector, 2100 E Elliot Rd, MD-EL725/Tempe//AZ/85284 (REPRINT); Motorola Inc, Semicond Prod Sector, Tempe//AZ/85284
 Journal: JOURNAL OF ELECTRONIC MATERIALS, 2001, V30, N8 (AUG), P1035-1041
 ISSN: 0361-5235 Publication date: 20010800
 Publisher: MINERALS METALS MATERIALS SOC, 184 THORN HILL RD, WARRENDALE, PA 15086 USA

Language: English Document Type: ARTICLE

Abstract: There are growing concerns in the electronics industry for not only finding alternatives to lead but also other potentially hazardous materials as well. This paper summarizes the development of ethylene **glycol** ether (EGE)-free solder flux for the formulation of lead-free solder pastes. Replacing the toxic components in the flux was only the first challenge, the criteria of commercially proven pastes also had to be met. Both commercial and in-house solder paste formulations were evaluated for printability, reflow, wetting, flux residue removal, and solder void characteristics. Two critical issues, solder bump voids and flux residue removal, were identified and associated with the high temperature reflow of Pb-free pastes. These issues were not effectively improved by the existing commercial EGE-free solder pastes. New solder paste formulations were developed utilizing alternative chemistry than those found in traditional solder paste fluxes. These pastes, some of which are also water soluble, reduced void frequency and size by more than 4x as compared to vendors' pastes. Solder bump height uniformity of 135 +/- 4 μ m within each **die** was consistently achieved. Thermal-mechanical reliability tests were performed on various lead-free solder alloys using the new flux formulations. The reliability of flip **chip** assembled DCA on organic boards with both OSP/Cu and Cu/Ni/Au **pad** metallizations were comparable to eutectic Sn63Pb37 bumped assemblies using commercial pastes.

29/3,AB/13 (Item 3 from file: 34)
 DIALOG(R)File 34:SciSearch(R) Cited Ref Sci
 (c) 2003 Inst for Sci Info. All rts. reserv.

09736461 Genuine Article#: 443LY Number of References: 35
 Title: Determination of metal-EDTA complexes in soil solution and plant xylem by ion chromatography-electrospray mass spectrometry (ABSTRACT AVAILABLE)

Author(s): Collins RN (REPRINT) ; Onisko BC; McLaughlin MJ; Merrington G
 Corporate Source: Univ Adelaide, Dept Soil & Water, Waite Campus/Glen
 Osmond/SA 5064/Australia/ (REPRINT); Univ Adelaide, Dept Soil &
 Water, Glen Osmond/SA 5064/Australia/; Zeneca Agr Prod, Western Reg
 Ctr, Richmond//CA/94804; CSIRO, Glen Osmond/SA 5064/Australia/
 Journal: ENVIRONMENTAL SCIENCE & TECHNOLOGY, 2001, V35, N12 (JUN 15), P
 2589-2593

ISSN: 0013-936X Publication date: 20010615

Publisher: AMER CHEMICAL SOC, 1155 16TH ST, NW, WASHINGTON, DC 20036 USA

Language: English Document Type: ARTICLE

Abstract: An ion chromatography-electrospray mass spectrometry (IC-MS) method was developed to quantify the metal complexes of ethylenediaminetetraacetic acid (EDTA) in soil solution and plant xylem exudate. Suitable separation of the metal-EDTA complexes was achieved on a Dionex AS5 column using 2 mM Na₂CO₃ as the eluant. However, satisfactory detection by eluant suppressed IC-MS, in either the positive or negative ion detection mode, could not be attained. A new eluant that still attained suitable separation and produced ionic species that could be detected by MS in the negative ion mode was developed. The eluant consisted of 2.5 mM (NH₄)₂CO₃, 9.7 mM NH₄OH, and 4% (v/v) methanol acid had a pH 9.9. Even though eluant suppressed IC-MS degraded detection limits by a factor of 4 over the nonsuppressed system, using the retention time and not the m/z (mass-to-charge ratio) of the intact chelate for identification, the latter allowed the metal complexes to be detected intact and was optimized for the analysis of environmental samples. The number of metal-EDTA species that could be detected was limited by the eluant used for ion chromatography (i.e. only those complexes that were stable at high pH), with metal-EDTA complexes of Al, Cd, Cu, Co, Mn, Ni, Pb, and Zn being adequately resolved. Iron(III), Ca, MgEDTA, and EDTA itself were not detected. Detection limits for the various complexes ranged from 0.1 to 1 µM.

29/3, AB/14 (Item 4 from file: 34)
 DIALOG(R) File 34:SciSearch(R) Cited Ref Sci
 (c) 2003 Inst for Sci Info. All rts. reserv.

07040303 Genuine Article#: 117FT Number of References: 39
 Title: Interaction of [(dien)Cu(ONO₂)]NO₃ and [(dien)Cu(Br)]Br with guanosine and adenine. Crystal structure of [(dien)Cu(Guo-N-7)(ONO₂)]NO₃ (ABSTRACT AVAILABLE)
 Author(s): Bolos CA (REPRINT) ; Christidis PC; Triantafyllou ST
 Corporate Source: ARISTOTELIAN UNIV THESSALONIKI, INORGAN CHEM LAB, DEPT GEN & INORGAN CHEM/THESSALONIKI 54006//GREECE/ (REPRINT); ARISTOTELIAN UNIV THESSALONIKI, APPL PHYS LAB/THESSALONIKI 54006//GREECE/
 Journal: POLYHEDRON, 1998, V17, N16, P2719-2726
 ISSN: 0277-5387 Publication date: 19980000
 Publisher: PERGAMON-ELSEVIER SCIENCE LTD, THE BOULEVARD, LANGFORD LANE, KIDLINGTON, OXFORD OX5 1GB, ENGLAND
 Language: English Document Type: ARTICLE
 Abstract: The reaction of [(dien)Cu(ONO₂)]NO₃ and [(dien)CuBr]Br with Guanosine(Guo) gave the compounds [(dien)Cu(Guo-N-7)(ONO₂)]NO₃ [dien = diethylenetriamine and [(dien)Cu(Guo)(Br)]Br, respectively. The reaction of [(dien)Cu(ONO₂)]NO₃ with Adenine(A) afforded the compound [(dien)Cu(A)(ONO₂)]NO₃. The complexes have been characterized by elemental analyses, molar conductance and magnetic measurements, as well as by electronic and IR spectral studies. According to the spectral data the ligands (Guo and A) bind to the copper atom via N-7. The crystal structure of the [(dien)Cu(Guo-N-7)(ONO₂)]NO₃ complex has been

determined by X-ray diffraction. Copper(II) is five coordinated with three nitrogen atoms from the **dien** molecule [2.015(6) Angstrom, 2.007(4) Angstrom, 2.038(6) Angstrom], the N-7 atom from guanosine [2.006(4) Angstrom] and an oxygen atom from a nitrate group [2.251(6) Angstrom]. The five donor atoms form a distorted square pyramid with the four nitrogen atoms on its basal plane and the oxygen atom at the apical position. All the complexes are electrolytes as indicated by their molar conductance in **methanol** or water. The magnetic moments are consistent with monomeric copper(II) units. (C) 1998 Elsevier Science Ltd. All rights reserved.

29/3,AB/15 (Item 5 from file: 34)
 DIALOG(R)File 34:SciSearch(R) Cited Ref Sci
 (c) 2003 Inst for Sci Info. All rts. reserv.

06431120 Genuine Article#: YR993 Number of References: 9
 Title: The dissolution of copper in common solvents used for low dielectric - polymers (ABSTRACT AVAILABLE)
 Author(s): Purdy AP (REPRINT) ; Godbey D; Buckley L
 Corporate Source: USN, RES LAB, MAT CHEM BRANCH, DIV CHEM, CODE 6120/WASHINGTON//DC/20375 (REPRINT)
 Journal: THIN SOLID FILMS, 1997, V308 (OCT '31), P486-489
 ISSN: 0040-6090 Publication date: 19971031
 Publisher: ELSEVIER SCIENCE SA LAUSANNE, PO BOX 564, 1001 LAUSANNE, SWITZERLAND

Language: English Document Type: ARTICLE

Abstract: A major issue with **copper metallization** is the diffusion and/or dissolution of copper into polymers used as insulating dielectric layers in **microelectronic** devices. Polymers are typically deposited by spin-coating. But when substrate-polymer interactions are studied, the process by which the polymer is deposited must be carefully considered. For example, what roles do the solvent or the curing conditions play in bringing copper into the polymer film? In this study, we focus on a frequently neglected aspect of the problem - that of solvent interactions with the copper. We have examined the ability of commonly used solvents to dissolve copper under oxygen atmospheres. For this **study**, **copper** turnings were stirred with the pure solvent under O-2. The presence of copper in solution was determined with spot tests, and solid reaction products were characterized with X-ray powder diffraction. Copper was found to dissolve in 1-methyl-2-pyrrolidinone, anisole, methylisobutylketone, propylene **glycol** methyl ether acetate, gamma-butyrolactone, dimethylacetamide, water, methylmethacrylate, m-cresol, and sulfolane in the presence of oxygen, but not in toluene, chlorobenzene, o-dichlorobenzene, chloroform, acetone, ethylacetate and tetrahydrofuran. (C) 1997 Elsevier Science S.A.

29/3,AB/16 (Item 6 from file: 34)
 DIALOG(R)File 34:SciSearch(R) Cited Ref Sci
 (c) 2003 Inst for Sci Info. All rts. reserv.

03902891 Genuine Article#: QQ510 Number of References: 29
 Title: EMBRYOTOXICITY OF SILVER IONS IS DIMINISHED BY CERULOPLASMIN - FURTHER EVIDENCE FOR ITS ROLE IN THE TRANSPORT OF COPPER (Abstract Available)
 Author(s): SHAVLOVSKI MM; CHEBOTAR NA; KONOPISTSEVA LA; ZAKHAROVA ET; KACHOURIN AM; VASSILIEV VB; GAITSKHOKI VS
 Corporate Source: RUSSIAN ACAD MED SCI, INST EXPTL MED, DEPT MOLEC GENET, 12 PAVLOV ST/ST PETERSBURG 197376//RUSSIA/; RUSSIAN ACAD MED SCI, INST

EXPTL MED, DEPT MOLEC GENET/ST PETERSBURG 197376//RUSSIA//; RUSSIAN ACAD
SCI, INST NUCL PHYS/GATCHINA 188350//RUSSIA/
Journal: BIOMETALS, 1995, V8, N2 (APR), P122-128
ISSN: 0966-0844
Language: ENGLISH Document Type: ARTICLE

Abstract: The effect of alimentary administration of silver salts upon embryogenesis in rats was studied. Feeding of female rats throughout the term on a regular **diet** supplemented with AgCl did not cause alterations of their physiological functions, despite the fact that enzymatically active copper-containing ceruloplasmin (CP) was eliminated from the blood plasma. However, developmental abnormalities of embryos, their prenatal death or the 100% mortality of the newborns in the first 24 h of life was seen. Copper content in placenta and fetal tissues was strongly diminished. Cu, Zn-superoxide dismutase (SOD) activity decreased in cytoplasm of embryonic cells along with a drop, though less pronounced, in the tissues of the pregnant females. Embryotoxicity of AgCl was seriously diminished by repetitive injections of native CP to the pregnant rats. Such treatment resulted in an increase of SOD activity in placenta and embryonic tissues. The mortality of the newborns also became less. It is suggested that the embryotoxic effect of AgCl is caused by its ability to interfere with copper metabolism, in particular by altering the copper-transporting function of CP.

29/3, AB/17 (Item 7 from file: 34)
DIALOG(R) File 34: SciSearch(R) Cited Ref Sci
(c) 2003 Inst for Sci Info. All rts. reserv.

03841883 Genuine Article#: QL285 Number of References: 57
Title: STRUCTURE AND REACTIVITY OF COPPER-ZINC-CADMIUM CHROMITE CATALYSTS
(Abstract Available)
Author(s): CASTIGLIONI GL; VACCARI A; FIERRO G; INVERSI M; JACONO ML; MINELLI G; PETTITI I; PORTA P; GAZZANO M
Corporate Source: UNIV BOLOGNA, DIPARTIMENTO CHIM IND & MAT, VIALE RISORGIMENTO 4/I-40136 BOLOGNA//ITALY//; UNIV BOLOGNA, DIPARTIMENTO CHIM IND & MAT/I-40136 BOLOGNA//ITALY//; UNIV ROMA LA SAPIENZA, CNR, CTR STRUTTURA & ATT CATALIT SISTEMI OSSIDI, DIPARTIMENTO CHIM/I-00185 ROME//ITALY//; CNR, CTR STRUTTURA & FIS MACROMOLEC, DIPARTIMENTO CHIM G CIAMICIAN/I-40127 BOLOGNA//ITALY/
Journal: APPLIED CATALYSIS A-GENERAL, 1995, V123, N1 (MAR 2), P123-144
ISSN: 0926-860X
Language: ENGLISH Document Type: ARTICLE

Abstract: Different mixed oxides containing Cu, Zn, Cd and Cr were obtained by heating hydroxycarbonate precursors at various temperatures and in different atmospheres, and characterized by using X-ray diffraction (XRD), infrared, diffuse reflectance spectroscopy, thermogravimetry, and BET and copper surface area analysis. The reduction process and the phase evolution during reduction of the mixed oxides was followed by temperature-programmed reduction and XRD. The mixed oxides were activated in an H-2/N-2 flow and then tested as catalysts in the vapour-phase hydrogenation of gamma-butyrolactone (GEL) and a solution of maleic anhydride (MA) in GEL (60:40 w/w). Depending on composition, heating temperature and atmosphere, the samples contained different phases, such as CuO, CdO, Cr2O3, CuCrO2, CuCrO4, alpha-CdCrO4, cubic and tetragonal CuCr2O4, and cubic Cu-Zn and Cu-Cd spinel-type phases. The partial substitution of Cu2+ ions with Zn2+ or Cd2+ ions stabilizes the cubic form of the spinel-type phases, which form via intermediate chromate phases. The final product of reduction in all cases is **metallic copper**. Cubic CuCr2O4 is more reducible than the corresponding tetragonal phase, which reduces to **metallic**

copper through the intermediate formation of CuCrO_2 . The presence of zinc or cadmium promotes or inhibits, respectively, the copper reducibility both in CuO and in the copper chromite spinel. In the hydrogenation of GBL, the Cu/Cr catalysts obtained by calcination show similar behaviours, with a small increase in activity as a function of the copper content. At the lower temperatures investigated the main products are tetrahydrofuran (THF) and n-butanol, while at 548 K significant amounts of ethanol are observed, favoured by increasing copper content. On the other hand, the Cu/Cr catalyst obtained by heating under a reduced oxygen atmosphere, for which only tetragonal CuCr_2O_4 is detected before reduction, shows a decrease in catalytic activity. Partial substitution of the Cu^{2+} ions gives rise to a decrease in GEL conversion, with a considerable deactivation when Cd^{2+} ions are present. Decreasing the H₂/C₄ molar ratio gives rise to a decrease in activity for all samples, without any significant change in the trend observed. With the MA/GBL solution, the partial substitution of Cd^{2+} ions for Cu^{2+} ions also gives rise to considerable deactivation. For the other catalysts, the main products at the lower temperatures are GEL and succinic anhydride (SA), while at 548 K overhydrogenation and hydrogenolysis reactions predominate. The presence of Zn^{2+} ions gives rise to an increase in yield in GEL, due to an increase in the hydrogenation activity toward SA, but the consecutive transformation of GEL at high temperature to low cost products is not inhibited. On the other hand, the presence in the Cu/Cr catalyst of only CuCr_2O_4 increases the yield in GEL, as a consequence of a strong inhibition of the overhydrogenation and/or hydrogenolysis reactions of GEL. With the MA/GBL solution, similar behaviours are observed when the H₂/C₄ molar ratio decreases. At the same time, however, the lack in the carbon balance increases and a displacement towards high temperature of the maximum yield in GEL is found.

29/3,AB/18 (Item 1 from file: 434)
 DIALOG(R)File 434:SciSearch(R) Cited Ref Sci
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08202899 Genuine Article#: J1449 Number of References: 26
 Title: STRUCTURE AND MAGNETISM OF ANALOGOUS O-BRIDGED (PHENOLATO) AND S-BRIDGED (THIOPHENOLATO) BINUCLEAR COPPER(II) COMPLEXES - CRYSTAL-STRUCTURE OF THE BINUCLEAR S-BRIDGED COMPLEX
 $\text{MU}-[2,6\text{-BIS}(4'\text{-CYCLOHEXYL-4'-HYDROXY-2',3'-DIAZABUTA-1',3'-DIEN-1'-YL)-4\text{-METHYLTHIOPHENOLATO}(3-)-\text{S},\text{N}_2,\text{O}(\text{CuI},2)]\text{-METHANOL-}\mu\text{-PYRAZOLYL-N,N')\text{-DICOPPER(II)}\text{S}$
 Author(s): ILIOPOULOS P; MURRAY KS; ROBSON R; WILSON J; WILLIAMS GA
 Corporate Source: MONASH UNIV,DEPT CHEM/CLAYTON/VIC 3168/AUSTRALIA/; UNIV MELBOURNE,DEPT INORGAN CHEM/PARKVILLE/VIC 3052/AUSTRALIA/; AUSTRALIAN RADIAT LAB/YALLAMBIE/VIC 3085/AUSTRALIA/
 Journal: JOURNAL OF THE CHEMICAL SOCIETY-DALTON TRANSACTIONS, 1987, N7, P 1585-1591
 Language: ENGLISH Document Type: ARTICLE

29/3,AB/19 (Item 1 from file: 94)
 DIALOG(R)File 94:JICST-EPlus
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04781482 JICST ACCESSION NUMBER: 01A0126727 FILE SEGMENT: JICST-E
 Electrodeposition of Void-free Copper from Ethylenediamine Complex Bath for ULSI Metallization.
 NAWAFUNE HIDEMI (1); MIZUMOTO SHOZO (1); KITAMURA HIROSHI (2); UCHIDA EI (3); OKADA TAKASHI (3)

(1) Konandai Haitekurisachise; (2) Konandai Daigakuinshizenkagakukenkkyuka
; (3) Ishihara Chem. Co., Ltd.

Hyomen Gijutsu(Journal of the Surface Finishing Society of Japan), 2000,
VOL.51,NO.11, PAGE.1142-1147, FIG.10, TBL.3, REF.11

JOURNAL NUMBER: G0441BAW ISSN NO: 0915-1869

UNIVERSAL DECIMAL CLASSIFICATION: 621.793.3+621.357.7 621.382.002.2

LANGUAGE: Japanese COUNTRY OF PUBLICATION: Japan

DOCUMENT TYPE: Journal

ARTICLE TYPE: Original paper

MEDIA TYPE: Printed Publication

ABSTRACT: Copper appears to offer better conductivity and reliability in ULSI metallization than aluminum, and the current research confirms copper potential in this regard. ULSI metallization is accomplished by electrodeposition of copper from ethylenediamine complex baths using the additives glycine, ammonium sulfate, and a small quantity of thiodiglycolic acid. These baths produce good throwing powder and fill trenches and contact holes on silicon **wafers** with giant grains of void-free **copper** deposits. ULSI **metallization** using **copper** produced no voids as are formed due to the absorption of polyethylene **glycol** in the presence of chloride ions, thus avoiding a major drawback of ULSI metallization. Results of the current research have demonstrated the potential for use of **copper** in ULSI **metallization** accomplished through electrodeposition of copper using ethylenediamine complex baths. (author abst.)

29/3,AB/20 (Item 2 from file: 94)

DIALOG(R)File 94:JICST-EPlus

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04615782 JICST ACCESSION NUMBER: 00A0442606 FILE SEGMENT: JICST-E
Fundamental **Study** on Electroless **Copper** Plating Bath Applied
for ULSI Wiring.

MATSUOKA M (1); ODANI M (1); TANIWAKI M (1); KAMO Y (1); TAMAKI J (1);
YAMAMOTO Y (1); IZUMI N (2); KAMISAWA A (2)

(1) Ritsumeikan Univ.; (2) Rohm Co., Ltd.

Ritsumeikan Daigaku Rikogaku Kenkyujo Kiyo(Memoirs of the Institute of
Science & Engineering, Ritsumeikan University), 2000, NO.58, PAGE.35-41
, FIG.7, REF.9

JOURNAL NUMBER: F0257AAJ ISSN NO: 0370-4254 CODEN: RDRKA

UNIVERSAL DECIMAL CLASSIFICATION: 621.793.3 621.3.049.77

LANGUAGE: English COUNTRY OF PUBLICATION: Japan

DOCUMENT TYPE: Journal

ARTICLE TYPE: Original paper

MEDIA TYPE: Printed Publication

ABSTRACT: As a novel wiring method, electroless **copper**
metallization attracts intensive attention to produce high
reliability and high density interconnection of submicron feature.
Electroless copper plating has a high potentiality for a uniform
filling of complicated trenches and vias. These features of electroless
plating are excellent as compared with electroplating and chemical
vapor deposition(CVD). In practical use of electroless copper plating,
adhesion, resistivity, internal stress and crystal structure of the
deposit are important factors. In this study, effect of chemical
composition of electroless copper solution and operating conditions on
the properties of the deposits was systematically investigated. As the
selective deposition of copper only on barrier surface such as TiN is
another key technology in fabricating ULSI(ultra large scale
integration) circuit, the selectivity of copper deposition is also
examined at different dissolved oxygen levels. Internal stress was
reduced by using low-pH plating solution and 2,2'-bipyridyl. Addition

of polyethylene **glycol**(PEG) functioning as a surfactant gave a favorable effect on coating property unless it was codeposited in the film. The selective seeding of Pd and Ag was achieved by using a solution based on nitric acid. Fine circuit patterns on silicon **wafer** were metallized uniformly by reducing dissolved oxygen level in electroless copper solution. (author abst.)

29/3,AB/21 (Item 3 from file: 94)
 DIALOG(R)File 94:JICST-EPlus
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03220509 JICST ACCESSION NUMBER: 97A0562952 FILE SEGMENT: JICST-E
 An Examination of Slurry for Wiring Metal's Chemical Mechanical Polishing.
 SAKAI KENJI (1); DOI TOSHIRO (2); KASAI TOSHIO (3)
 (1) Tokyo Seimitsu Co., Ltd.; (2) Saitama Univ., Fac. of Educ.; (3) Saitama Univ., Fac. of Eng.
 Toryu Kako Gakkaishi(Journal of the Japan Society of Grinding Engineers),
 1997, VOL.41,NO.6, PAGE.231-233, FIG.4, -TBL.2-
 JOURNAL NUMBER: L0473AAD ISSN NO: 0914-2703
 UNIVERSAL DECIMAL CLASSIFICATION: 621.92 .621.382.002.2
 LANGUAGE: Japanese COUNTRY OF PUBLICATION: Japan
 DOCUMENT TYPE: Journal
 ARTICLE TYPE: Original paper
 MEDIA TYPE: Printed Publication

ABSTRACT: Since the finishing agents (slurry) for planarization polishing of the current LSI devices all contain abrasive grains, they produce short life, rust on machines to give unsatisfactory finished surface and difficulty in cleaning. A mixed slurry of nitric acid, hydrogen peroxide and polyethylene **glycol** with conventional machining performance without abrasive grains by examining the planarization polishing characteristics using the wiring **metal Cu** as the object. The machined surface of higher quality than that with Al₂O₃-based abrasive grain containing slurry was attained with machining speed of 0.3.MU.m/min and surface roughness of 40-80.ANGS.Rmax.

29/3,AB/22 (Item 1 from file: 99)
 DIALOG(R)File 99:Wilson Appl. Sci & Tech Abs
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2401611 H.W. WILSON RECORD NUMBER: BAST01102752
 Ethylene **glycol** ether free solder paste development
 Lytle, Bill; Fang, Treliant; Li, Li
 Journal of Electronic Materials v. 30 no8 (Aug. 2001) p. 1035-41
 DOCUMENT TYPE: Feature Article ISSN: 0361-5235

ABSTRACT: There are growing concerns in the electronics industry for not only finding alternatives to lead but also other potentially hazardous materials as well. This paper summarizes the development of ethylene **glycol** ether (EGE)-free solder flux for the formulation of lead-free solder pastes. Replacing the toxic components in the flux was only the first challenge, the criteria of commercially proven pastes also had to be met. Both commercial and in-house solder paste formulations were evaluated for printability, reflow, wetting, flux residue removal, and solder void characteristics. Two critical issues, solder bump voids and flux residue removal, were identified and associated with the high temperature reflow of Pb-free pastes. These issues were not effectively improved by the existing commercial EGE-free solder pastes. New solder paste formulations were developed utilizing alternative chemistry than those found in traditional

solder paste fluxes. These pastes, some of which are also water soluble, reduced void frequency and size by more than 4x as compared to vendors' pastes. Solder bump height uniformity of 135 [plus or minus] 4 μ m within each die was consistently achieved. Thermal-mechanical reliability tests were performed on various lead-free solder alloys using the new flux formulations. The reliability of flip chip assembled DCA on organic boards with both OSP/Cu and Cu/Ni/Au pad metallizations were comparable to eutectic Sn63Pb37 bumped assemblies using commercial pastes. Reprinted by permission of the publisher.

29/3,AB/23 (Item 1 from file: 144)
 DIALOG(R)File 144:Pascal
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15893583 PASCAL No.: 03-0031936
 Investigation of Carrying Agents on Microstructure of Electroplated Cu Films

SHIEH Jia-Min; CHANG Shih-Chieh; DAI Bau-Tong; FENG Ming-Shiann
 National Nano Device Laboratories, Hsinchu 30050, Taiwan, R.O.C.;
 Institute of Materials Science and Engineering, National Chiao Tung University, Hsinchu 30050, Taiwan, R.O.C.

Journal: Japanese Journal of Applied Physics, Part I : Regular papers, short notes & review papers, 2002-11, 41 (11A) 6347-6350

Language: English

Adding high molecular-weight polyethylene glycol (PEG) as a carrying agent benefits Cu electroplating from the viewpoint of an increase in both filling capability and films<right single quotation mark> conductivity, when plated in a lower current-density region. On electroplating in a higher current-density region, a gradually decreasing grain size from the top to bottom of Cu films occurs in the electrolyte without PEG or with PEG200. In comparison, sharp grains are formed for the mentioned multi-domain crystallization for those films deposited by the electrolyte containing PEG2000 or PEG6000 due to its lower surface tension which facilitates grain growth along the higher electric field. The used concentration of PEG was flexible due to the fact that the surface morphology, resistivity and deposition rate of deposited films remained almost unchanged over a wide PEG concentration range. (c) 2002 The Japan Society of Applied Physics

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29/3,AB/24 (Item 2 from file: 144)
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15424750 PASCAL No.: 02-0116455
 Analysis of current-potential hysteresis during electrodeposition of copper with additives

HEBERT Kurt R; Z

Department of Chemical Engineering, Iowa State University, Ames, Iowa 50011, United States

Journal: Journal of the Electrochemical Society, 2001, 148 (11) C726-C732

Language: English

Two models are described for cyclic voltammetry during electrodeposition of copper from an acid sulfate bath containing the additives polyethylene glycol (PEG), chloride ions and mercapto-1-propanesulfonate (MPSA). The same bath may be used for superfilling of cavities during fabrication of copper on-chip metallization. Experimental

current-potential scans show a characteristic hysteresis in solutions with all three additives, which demonstrates the presence of both activated and inhibited kinetics. Both models assume that deposition is inhibited by a PEG-chloride adsorbed surface complex, as established previously. One model further hypothesizes that PEG is incorporated into grain boundaries in the copper at the same fractional coverage found on the external surface. The second model neglects PEG incorporation, but assumes that adsorbing MPSA directly displaces PEG from the surface. Both models predict hysteresis quantitatively similar to experiments, without the use of fitting parameters. The competitive adsorption model is favored because, unlike the PEG incorporation model, it yields realistic predictions of carbon and sulfur concentrations in the deposit.

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29/3,AB/25 (Item 3 from file: 144)
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15276516 PASCAL No.: 01-0446946
 Ethylene glycol ether free solder paste development
 LYTLE Bill; TRELIANT FANG; LI LI; ZHANG Charles
 Motorola, Semiconductor Products Sector, 2100 East Elliot Road, MD:EL725,
 Tempe, AZ 85284, United States
 Journal: Journal of electronic materials, 2001, 30 (8) 1035-1041
 Language: English
 There are growing concerns in the electronics industry for not only finding alternatives to lead but also other potentially hazardous materials as well. This paper summarizes the development of ethylene glycol ether (EGE)-free solder flux for the formulation of lead-free solder pastes. Replacing the toxic components in the flux was only the first challenge, the criteria of commercially proven pastes also had to be met. Both commercial and in-house solder paste formulations were evaluated for printability, reflow, wetting, flux residue removal, and solder void characteristics. Two critical issues, solder bump voids and flux residue removal, were identified and associated with the high temperature reflow of Pb-free pastes. These issues were not effectively improved by the existing commercial EGE-free solder pastes. New solder paste formulations were developed utilizing alternative chemistry than those found in traditional solder paste fluxes. These pastes, some of which are also water soluble, reduced void frequency and size by more than 4x as compared to vendors' pastes. Solder bump height uniformity of 135 +/- 4 μ m within each die was consistently achieved. Thermal-mechanical reliability tests were performed on various lead-free solder alloys using the new flux formulations. The reliability of flip chip assembled DCA on organic boards with both OSP/Cu and Cu/Ni/Au pad metallizations were comparable to eutectic Sn63Pb37 bumped assemblies using commercial pastes.

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29/3,AB/26 (Item 4 from file: 144)
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14978576 PASCAL No.: 01-0132203
 En Japonais
 (Electrodeposition of void-free copper from ethylenediamine complex bath for ULSI metallization)
 NAWAFUNE Hidemi; KITAMURA Hiroshi; MIZUMOTO Shozo; UCHIDA Ei; OKADA

Takashi

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Japan; ISIHARA Chemical Co., Ltd., 5-26, Nishiyanagihara-cho, Hyogo-ku,
Kobe-shi, Hyogo 652-0806, Japan

Journal: Hyomen gijutsu, 2000, 51 (11) 1142-1147

Language: Japanese Summary Language: English

Cooper appears to offer better conductivity and reliability in ULSI metallization than aluminum, and the current research confirms copper potential in this regard. ULSI metallization is accomplished by electrodepositing copper from ethylenediamine complex baths using the additives glycine, ammonium sulfate, and a small quantity of thiodiglycolic acid. These baths produce good throwing powder and fill trenches and contact holes on silicon wafers with giant grains of void-free copper deposits. ULSI metallization using copper produced no voids as are formed due to the absorption of polyethylene glycol in the presence of chloride ions, thus avoiding a major drawback of ULSI metallization. Results of the current research have demonstrated the potential for use of copper in ULSI metallization accomplished through electrodeposition of copper using ethylenediamine complex baths.

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29/3,AB/27 (Item 5 from file: 144)
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14962749 PASCAL No.: 01-0115661

Charge modulated electrochemical deposition of copper for electronic interconnect applications

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Faraday Technology, Inc., 315 Huls Dr., Clayton, OH 45315, United States

Journal: Plating and surface finishing, 2000, 87 (12) 68-73

Language: English

This paper describes the application of "charge-modulated electrodeposition" (CM-ECD) for copper plating of z-axis interconnects for electronic applications. Instead of brighteners, levelers and/or suppressors (i.e. chemical mediation), CM-ECD uses a charge-modulated waveform to control the current distribution and therefore the copper distribution within the interconnect. By tuning the CM-ECD process parameters, electrical mediation of copper deposition of interconnects ranging from plated-through-holes (PTHs) for printed wiring board (PWB) applications to microvias for high density interconnect (HDI) substrates to sub-micron trenches for integrated circuits (IC) is accomplished.

Data are presented for copper plating of (1) 325 μm PTHs, (2) 100 μm microvias and (3) 0.25 to 1 μm trenches for IC applications, using a simple copper sulfate/sulfuric acid plating bath containing chloride and polyethylene glycol. In addition, plating of PTHs and microvias on the same substrate is accomplished by sequentially tuning the CM-ECD process, without masking.

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29/3,AB/28 (Item 6 from file: 144)
DIALOG(R)File 144:Pascal
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14910014 PASCAL No.: 01-0059472

Superconformal electrodeposition of copper in 500-90 nm features

MOFFAT T P; BONEVICH J E; HUBER W H; STANISHEVSKY A; KELLY D R; STAFFORD G R; JOSELL D

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Journal: Journal of the Electrochemical Society, 2000, 147 (12) 4524-4535

Language: English

Superconformal electrodeposition of copper in 500 nm deep trenches ranging from 500 to 90 nm in width has been demonstrated using an acid cupric sulfate electrolyte containing chloride (Cl), polyethylene glycol (PEG), and 3-mercapto-1-propanesulfonate (MPSA). In contrast, similar experiments using either an additive-free electrolyte or an electrolyte containing the binary combinations Cl-PEG, Cl-MPSA, or simply benzotriazole (BTAH), resulted in the formation of a continuous void within the center of the trench. Void formation in the latter electrolytes is shown to be reduced through the geometrical leveling effect associated with conformal deposition in trenches or vias with sloping sidewalls. The slanted sidewalls also counterbalance the influence of the differential cupric ion concentration that develops within the trenches. Examination of the i-E deposition characteristics of the electrolytes reveals a hysteretic response associated with the Cl-PEG-MPSA electrolyte that can be usefully employed to monitor and explore additive efficacy and consumption. Likewise, resistivity measurements performed on corresponding blanket films can be used to quantify the extent of additive incorporation and its influence on microstructural evolution. The films deposited from the Cl-PEG-MPSA electrolyte exhibit spontaneous recrystallization at room temperature that results in a 23% drop in resistivity within a few hours of deposition.

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29/3,AB/29 (Item 7 from file: 144)

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14445688 PASCAL No.: 00-0104377

Copper electroplating for future ultralarge scale integration interconnection

GAU W C; CHANG T C; LIN Y S; HU J C; CHEN L J; CHANG C Y; CHENG C L

Department of Chemical Engineering, Chung-Yuan University, Chung-Li, Taiwan, Republic of China; National Nano Device Laboratory, Hsin-Chu, Taiwan, Republic of China; Department of Physics, National Sun-Yat-Sen University, Kaohsiung, Taiwan, Republic of China; Department of Chemical Engineering, Chung-Yuan University, Chung-Li, Taiwan, Republic of China; Department of Materials Science and Engineering, National Tsing-Hua University, Hsin-Chu, Taiwan, Republic of China; Department of Electronics Engineering and Institute of Electronics, National Chiao-Tung University, Hsin-Chu, Taiwan, Republic of China; Department of Chemical Engineering, Chung-Yuan University, Chung-Li, Taiwan, Republic of China

Journal: Journal of vacuum science and technology. A. Vacuum, surfaces, and films, 2000-03, 18 (2) 656-660

Language: English

Copper has received considerable attention during the past few years because of its low electrical resistivity, high melting temperature, and

high electromigration resistance. Since electrochemical plating has several advantages compared to physical vapor deposition and chemical vapor deposition due to its low cost and low processing temperature, it becomes the most attractive technique for the implementation of **Cu metallization**. In this article, we report an electroplating scheme for **Cu** deposition and **study** aspects of **copper** electroplating related to chemical additives effected on Cu deposition. A low resistivity Cu film (similar $2 \mu\Omega$ <hair thin space>cm) could be obtained when metallic ion concentration is decreased or current density is increased. The detailed correlation between film resistivity, electrolyte concentration, current density, and film morphology is given in this study. In addition, chemical additives in electrolyte solutions also play important roles in copper electroplating. In this report, thiourea and polyethylene **glycol**, which are usually added in printing circuit board electroplating to influence nucleation, have been used as gap filling promoters to help Cu filling. The results show that addition of thiourea and polyethylene **glycol** could help in forming smooth Cu film but does not promote Cu filling ability. Hydroxyl amine sulfate, which has both amino and sulfate groups, is proposed for use as a gap filling promoter in helping Cu electroplating. In this work, we demonstrate that Cu could be electroplated into fine trenches (at $0.3 \mu\text{m}$ dimension with aspect ratio of 3) when hydroxyl amine sulfate is present and no voids are formed. (c) 2000 American Vacuum Society.

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29/3,AB/30 (Item 8 from file: 144)
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13421568 PASCAL No.: 98-0115145

The dissolution of copper in common solvents used for low dielectric polymers

PURDY A P; GODBEY D; BUCKLEY L

Materials Chemistry Branch, Code 6120, Chemistry Division, Naval Research Laboratory, Washington, United States

ICMCTF 1997 Special issue

Journal: Thin solid films, 1997, 308-09 486-489

Language: English Summary Language: English

Copyright (c) 1997 Elsevier Science S.A. All rights reserved. A major issue with **copper metallization** is the diffusion and/or dissolution of copper into polymers used as insulating dielectric layers in **microelectronic** devices. Polymers are typically deposited by spin-coating. But when substrate-polymer interactions are studied, the process by which the polymer is deposited must be carefully considered. For example, what roles do the solvent or the curing conditions play in bringing copper into the polymer film? In this study, we focus on a frequently neglected aspect of the problem - that of solvent interactions with the copper. We have examined the ability of commonly used solvents to dissolve copper under oxygen atmospheres. For this **study**, **copper** turnings were stirred with the pure solvent under O_2 . The presence of copper in solution was determined with spot tests, and solid reaction products were characterized with X-ray powder diffraction. Copper was found to dissolve in 1-methyl-2-pyrrolidinone, anisole, methylisobutylketone, propylene **glycol** methyl ether acetate, gamma-butyrolactone, dimethylacetamide, water, methylmethacrylate, m-cresol, and sulfolane in the presence of oxygen, but not in toluene, chlorobenzene, dichlorobenzene, chloroform, acetone and tetrahydrofuran.

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29/3,AB/31 (Item 9 from file: 144)
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12304226 PASCAL No.: 95-0537828

In situ quadrupole mass spectroscopy studies of water and solvent coordination to copper(II) beta -diketonate precursors: implications for the chemical vapor deposition of copper

Copper metallization for future VLSI

ZHENG B; GOLDBERG C; EISENBRAUN E T; LIU J; KALOYEROS A E; TOSCANO P J;
 MURARKA S P; LOAN J F; SULLIVAN J

MURARKA Shyam P, ed; GUTMANN Ronald J, ed

SUNY Univ. Albany, New York state cent., dep. physics, Albany NY 12222,
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Rensselaer polytech. inst., cent. integrated electronics, Troy NY 12180,
 USA

Journal: Materials chemistry and physics, 1995, 41 (3) 173-181

Language: English

The incorporation of Cu(II) beta -diketonates in technologically useful chemical vapor deposition (CVD) processes requires identifying the nature of the coordinated complexes that result from variations in the methods used for precursor preparation, and exploring their effects on its stability and performance. Accordingly, in situ quadrupole mass spectrometry (QMS)) was employed to investigate the gas-phase evolution of various coordinated forms of CU SUP I SUP I (hfac) SUB 2 and CU SUP I SUP I (tdf) SUB 2 where tdf = 1,1,1,2,2,3,3,7,7,8,8,9,9,9-tetradecafluorononane-4,6-dione and hfac = hexafluoroacetylacetonate, to determine the nature of these coordinated complexes and elucidate the desorption energetics of associated additives. Careful analyses of complex fragmentation patterns during its sublimation and transport to the CVD reactor were performed under a variety of electron energies to develop an understanding of the effects of synthetic variations on precursor coordination, determine the fragmentation modes of the parent molecular ions and associated ionic fragments, and identify the mechanisms of precursor-additive interactions. In particular, it was found that Cu(hfac) SUB 2 coordinates with water, **methanol** , or ethanol, but not with 2-propanol. Additionally, QMS studies of Cu(hfac) SUB 2 seem to indicate that the use of **methanol** in the final recrystallization step involved in its synthesis yields a mixture of the coordinated complexes CU SUP I SUP I (hfac) SUB 2 .H SUB 2 O.CH SUB 3 OH, CU SUP I SUP I (hfac) SUB 2 .2H SUB 2 O, and CU SUP I SUP I (hfac) SUB 2 .2CH SUB 3 OH. Similarly, in the case of CU SUP I SUP I (tdf) SUB 2 , the use of ethanol and trichloromethane appears to produce a mixture of the coordinated complexes Cu SUP I SUP I (tdf) SUB 2 .C SUB 2 H SUB 5 OH.H SUB 2 O, Cu SUP I SUP I (tdf) SUB 2 .C SUB 2 H SUB 5 OH, and CU SUP I SUP I (tdf) SUB 2 2H SUB 2 O. By determining the temperature above which solvent molecules decoordinate from th

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11241181 PASCAL No.: 94-0059230

Effect of pretreatment conditions of Si-CuCl mixtures on the rate and selectivity in the reaction of silicon with **methanol** using copper(I) chloride catalyst

OKAMOTO M; OSAKA M; YAMAMOTO K I; SUZUKI E; ONO Y

Tokyo univ. technology, dep. chemical eng., Meguro-ku, Tokyo 152, Japan

Journal: Journal of catalysis, 1993, 143 (1) 64-85

Language: English

The reaction of silicon with **methanol** was carried out in a fixed-bed flow reactor using copper(I) chloride as a catalyst. The rate of the reaction and the selectivity for trimethoxysilane greatly depends on the preheating conditions of the Si-CuCl mixture prior to feeding **methanol**. When the preheating temperature was above 623 K, trimethoxysilane was formed with selectivities around 65%, the remainder being tetramethoxysilane. The XRD and EPMA studies revealed that the Cu SUB 3 Si phase was scattered on the silicon surface upon heating the Si-CuCl mixture above 623 K

29/3,AB/33 (Item 11 from file: 144)
DIALOG(R)File 144:Pascal
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08265767 PASCAL No.: 88-0266274
Corrosion in solar heating systems. Pt. 1:Copper behaviour in water/
glycol solutions

MONTICELLI C; BRUNORO G; TRABANELLI G; FRIGNANI A
Ferrara Univ. Ist. Chmico, Italy
Journal: Werkst. Korros., 1986-09, 37 (9) 479-484
Language: ENGLISH

Diese Arbeit enthaelt **die** experimentellen Ergebnisse ueber den Korrosionswiderstand von Kupfer in Ethylen- und Propylen-**Glycol**/Wasser-Gemischen, wie sie ueblicherweise als Waermetransportmedium benutzt werden. Es wurden langzeitige gravimetrische Tests an Elektrolytkupfer bei 80 C durchgefuehrt, auch in Glykol/Wasser-Gemischen, **die** vorher auf diese Temperatur erhitzt oder durch 200 ppm Chlorid verunreinigt worden waren. **Die** chemische Zusammensetzung, **die** Halbleitereigenschaft en sowie **die** Morphologie wurden durch Feinstrukturuntersuchungen, Pulsphotopotentialmessungen und mit dem Rasterelektronenmikroskop untersucht. Der Einfluss des Waermedurchgangs auf **die** Kupferkorrosion und den Elementstrom der Kombination Kupfer/6351 Aluminium wurde durch elektrochemische Messungen verfolgt. **Die** folgenden Ergebnisse wurden erhalten: Ethylen- und Propylen-**Glycol** /Wasser-Gemische haben eine geringe Aggressivitaet. Dennoch erhoehen Chloride, sowie eine teilweise Zersetzung des Glycols durch hohe Temperatur **die** Aggressivitaet erheblich. Unter allen experimentellen Bedingungen waren **die** Korrosionsraten des Kupfers in Ethylenglycolloesungen hoeher als in Prophylenglycolloesungen. In chloridfreien Medien stimuliert ein Waermedurchgang **die** kathodische Reaktion der Kupferkorrosion. Eine Elementbindung zwischen Kupfer und Aluminium fuehrt zu einer Lochkorrosion des Aluminiums. **Die** Lochkorrosion wird durch Waermetransport und Gegenwart von Chlorid in der Loesung beschleunigt, besonders in Ethylengly

29/3,AB/34 (Item 1 from file: 315)
DIALOG(R)File 315:ChemEng & Biotec Abs
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474321 CEABA Accession No.: 32-03-001221 DOCUMENT TYPE: Journal
Title: Report on the electrolytic industries for the year 1999
Orig. Title: Bericht ueber **die** Elektrochemische Industrie fuer das Jahr 1999

AUTHOR: Weidner, J.W. ; Doyle, M.

CORPORATE SOURCE: University of South Carolina, USA

JOURNAL: J. Electrochem. Soc., Volume: 147, Issue: 10, Page(s):
3953-3974

CODEN: JESOAN ISSN: 00134651

PUBLICATION DATE: 2000 (20000000) LANGUAGE: English

ABSTRACT: This report summarized information published on electrolytic

industries with particular attention to the chlor-alkali industry, chlorine without soda together with fluorine and fluorinated gases. The report went on to consider salt production, sodium chlorate, soda ash production and other sodium chemicals, metals, batteries and fuel cells. Particular focus was on production, plant capacities, consumption, demand, trends, prices, raw materials, new technology, and environmental issues.

29/3,AB/35 (Item 2 from file: 315)
 DIALOG(R)File 315:ChemEng & Biotec Abs
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448458 CEABA Accession No.: 29-12-020631 DOCUMENT TYPE: Journal
 Title: The partial oxidation of methane to **methanol**: an approach to catalyst design.
 AUTHOR: Taylor, S. H. ; Hargreaves, J. S. J. ; Hutchings, G. J. ; Joyner, R. W. ; Lembacher, C. W.
 CORPORATE SOURCE: Univ. Wales-Cardiff Dept. Chem. CF1 3TB UK
 CONFERENCE INFORMATION: Third workshop on XXX hydrocarbon conversion, Krasnoyarsk, Russia, 14-17 Jul 1997
 JOURNAL: Catal. Today, Volume: 42, Issue: 3, Page(s): 217-224
 CODEN: CATTEA ISSN: 09205861
 PUBLICATION DATE: 9 Jul 1998 (980709) LANGUAGE: English
 ABSTRACT: A new approach for the design of catalysts for methane partial oxidation to **methanol** is outlined, which involves identifying metal oxide catalyst components which activate methane and oxygen, but do not readily destroy **methanol**, the desired product. A series of catalysts based on molybdenum trioxide and tungsten trioxide were prepared by impregnation with solutions containing the metal ions of the second component. A copper/MoO₃ catalyst showed a selectivity advantage over homogeneous gas-phase oxidation in a reactor bed packed with quartz **chips**. In general, the WO₃-based catalysts were less effective for the production of **methanol**. Additionally, catalysts composed of physical mixtures of gallium(III) oxide or zinc oxide with MoO₃ were prepared. The Ga₂O₃/MoO₃ catalyst showed a maximum **methanol** yield greater than that of the homogeneous gas-phase reaction over an inert bed of quartz particles. The increased **methanol** yield was attributed to a synergistic effect.

29/3,AB/36 (Item 3 from file: 315)
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424453 CEABA Accession No.: 28-11-021829 DOCUMENT TYPE: Journal
 Title: Supercritical fluids that create and destroy.
 JOURNAL: Process Eng. (London), Volume: 78, Issue: 5, Page(s): 27
 CODEN: PSEGAP ISSN: 03701859
 PUBLICATION DATE: May 1997 (970500) LANGUAGE: English
 ABSTRACT: Researchers at the University of Bradford are using supercritical carbon dioxide to crystallize solutes as fine, dry particles. A **methanol**-saturated aqueous solutions of lactose is mixed with a stream of supercritical carbon dioxide in a high-pressure nozzle, causing the lactose to crystallize as a nest of dry, **wafer**-like, 3- μ m-diameter crystals, and simultaneously removing the solvents. Another group of researchers at the Slovenian National Institute of Chemistry is using supercritical water to oxidize acetic acid in the presence of a catalyst based on the mixture of cobalt, copper and zinc oxides. The catalysed oxidation reaction, which requires pressures of 240 bar and a temperature of 420.degree.C, has a

activation energy of 109.7 kJ/mol, compared with 180 kJ/mol for the noncatalytic reaction.

29/3,AB/37 (Item 4 from file: 315)
DIALOG(R)File 315:ChemEng & Biotec Abs
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222925 CEABA Accession No.: 19-12-015743 DOCUMENT TYPE: Journal
Title: Promotion of **methanol** synthesis and the water-gas shift reactions by adsorbed oxygen on supported copper catalysts
AUTHOR: Chinchin, G.C.; Spencer, M.S.; Waugh, K.C.; Whan, D.A.
JOURNAL: Journal of the Chemical Society, Faraday Transactions 1, Volume: 83, Issue: 7, Page(s): 2193-2212
CODEN: JCFTAR ISSN: 0300-959
PUBLICATION DATE: 1987 (870000) LANGUAGE: English
ABSTRACT: Trotz des industriellen Einsatzes von CuZnOAl₂O₃-Katalysatoren fuer die **Methanolsynthese** und die Wassergas-Shift-Reaktionen sind wichtige mechanistische Punkte umstritten. Hier werden die Reaktionen von CO + H₂, CO₂ + H₂ und CO₂ + CO + H₂ an polykristallinem Cu, dem industriellen Traegerkatalysator und CuAl₂O₃-Katalysatoren untersucht. Die Hauptquellen fuer den **Methanolkohlenstoff** bei der letztgenannten Gasmischung ist CO₂. Die kritischen Reaktionsschritte bei der Reaktion erfolgen an der **Cu-Metalloberflaeche**, es gibt keine synergistische Wirkung zwischen Cu und ZnO, letzteres wird demnach aus anderen Gruenden gebraucht. Unter den Reaktionsbedingungen entstehen mit CO₂ an Cu adsorbierte O-Atome wie bei der Katalysatoroxidation mit O₂ oder N₂O. Die Induktionsperiode fuer die **Methanolsynthese** entspricht der Bildung der Schicht der O-Atome, die bei beiden Reaktionen als Promotor und Zwischenprodukt noetig sind. Format ist nur bei der **Methanolsynthese** beteiligt. (Weckend)

29/3,AB/38 (Item 5 from file: 315)
DIALOG(R)File 315:ChemEng & Biotec Abs
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221428 CEABA Accession No.: 19-12-014246 DOCUMENT TYPE: Journal
Title: **Study** of Cu(2+)-doped zeolites Na-rho, K-rho, and Ca-rho by electron spin resonance and electron spin echo modulation spectroscopies
AUTHOR: Anderson, M.W.; Kevan, L.
JOURNAL: Journal of Physical Chemistry, Volume: 91, Issue: 11, Page(s): 2926-2930
CODEN: JPCHAX ISSN: 0022-365
PUBLICATION DATE: 1987 (870000) LANGUAGE: English
ABSTRACT: Die Mikroporen und die Ionenaustauscher-Eigenschaften machen Zeolithe zu besonders geeigneten hoch selektiven Katalysator-Substraten. Auf die Struktur des hydratisierten paramagnetischen Cu(2+) als katalytischem Zentrum im rho-Zeolithen und den Aktivierungsmechanismus haben die Gegenkationen (Na,K,Ca) wenig Einfluss - im Gegensatz zu deren Einfluss bei A-, X- und Y-Zeolithen. Wegen der Blockierung der Eingaenge der alpha-Kaefige durch die Gegenkationen koennen allerdings nichtpolare Molekuele wie Ethen nicht in die Zeolithe eindringen. Polare Molekuele wie NH₃, **Methanol** und Ethanol umgehen sie durch Solvation und komplexieren die Cu(2+)-Ionen. Durch sterische Hinderung ist die Zahl der komplexierenden Liganden aber kleiner als in H-rho-Zeolith. (Weckend)

29/3,AB/39 (Item 6 from file: 315)
 DIALOG(R)File 315:ChemEng & Biotec Abs
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202394 CEABA Accession No.: 18-12-015718 DOCUMENT TYPE: Journal
 Title: Electron spin resonance and electron spin echo **study** of
 Cu(2+) in zeolites H-rho and CsH-rho
 AUTHOR: Anderson, M.W.; Kevan, L.
 JOURNAL: Journal of Physical Chemistry, Volume: 90, Issue: 24, Page(s):
 6452-6459

CODEN: JPCHAX ISSN: 0022-365

PUBLICATION DATE: 1986 (860000) LANGUAGE: English

ABSTRACT: **Die** katalytische Aktivitaet von Zeolithen mit
 Uebergangsmetallen haengt von deren Lage im Gitter und der
 Wechselwirkung mit Adsorbaten ab. Cu(II)-Zeolithe sind Katalysatoren
 fuer **die** Oxidation von Propylen, **die** Cyclodimerisierung von
 Butadien und das Cracken von Cumen. Mit Elektronenspin echo-Spektroskopi-
 e lassen sich bei den paramagnetischen Cu(2+)-Kationen **die**
 Koordinationszahl und **die** Lage von Adsorbaten relativ zum
 paramagnetischen Zentrum bestimmen. Insbesondere werden hier **die**
 Echomodulationen durch Caesium und Deuterium verwendet. **Die**
 Cs-Modulation laesst sich simulieren, obwohl in diesem Fall nur ein
 Teil der Cs-Kationen dazu beitraegt, so dass **die** normale
 sphaeerische Durchschnittsbildung nicht angewendet werden kann.
Die Ergebnisse zeigen eine starke Affinitaet des Cu(2+) fuer
 Positionen in den alpha-Kaefigen auch nach der Dehydratation. Bei
 Adsorption erfolgt eine Wechselwirkung zwischen Cu(2+) und einem
 Ethen-Molekuel bzw. zwei **Methanol**-Molekuelen. (Weckend)

29/3,AB/40 (Item 7 from file: 315)
 DIALOG(R)File 315:ChemEng & Biotec Abs
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199893 CEABA Accession No.: 18-12-013217 DOCUMENT TYPE: Journal
 Title: Electron spin echo **study** of Cu(2+)-doped zeolite K-ZK4:
 Cation location and adsorbate interaction
 AUTHOR: Anderson, M.W.; Kevan, L.
 JOURNAL: Journal of Physical Chemistry, Volume: 90, Issue: 14, Page(s):
 3206-3212

CODEN: JPCHAX ISSN: 0022-365

PUBLICATION DATE: 1986 (860000) LANGUAGE: English

ABSTRACT: Ursache der Bedeutung der selektiven Zeolith-Katalysatoren sind
 ihre gut definierten Reaktionszentren und ihre Molekularsieb-Eigenschaf-
 ten. Zur gezielten Herstellung von Zeolithen mit bestimmten
 Eigenschaften ist **die** Kenntnis der Kationenstellen und der
 Wechselwirkung mit Adsorbaten wichtig. Bei den paramagnetischen
 Cu(2+)-Zentren in einer Reihe von K-ZK4-Zeolithen lassen sich **die**
 Koordination und **die** Geometrie dieser Kationen sowie der
 Einfluss des SiAl-Verhaeltnisses mit ESR bestimmen. **Die**
 Koordination ist oktaedrisch, trigonal-bipyramidal oder, wenn diese
 Komplexe geometrisch verhindert werden, tetraedrisch. Bei mehr als 10
 K(+)-Kationen pro Elementarzelle ist nur ein Wassermolekuel am Cu(2+)
 im alpha-Kaefig, im weniger haeufig besetzten beta-Kaefig sind es 3.
 Eine neue Cu(2+)-Art im alpha-Kaefig koordiniert 2 Wassermolekuele bei
 weniger als 10 K(+). Bei sehr geringem K(+)-Anteil gibt es einen
 grossen Komplex mit 3 **Methanol**-Molekuelen. Demnach befindet sich
 K(+) bevorzugt in den 6-Ringen, nicht in den 8-Ringen. (Weckend)

29/3,AB/41 (Item 8 from file: 315)
 DIALOG(R)File 315:ChemEng & Biotec Abs
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183488 CEABA Accession No.: 17-12-017546 DOCUMENT TYPE: Journal
 Title: Thin-layer chromatographic enantiomeric resolution via ligand exchange
 Orig. Title: Duennschicht-chromatographische Enantiomerentrennung mittels Ligandenaustausch
 AUTHOR: Guenther, K.
 JOURNAL: GIT-Supplement, Issue: 3, Page(s): 6-12
 CODEN: GITSD4 ISSN: 0930-406
 PUBLICATION DATE: 1986 (860000) LANGUAGE: German
 ABSTRACT: Neben den bisherigen Trenntechniken (GC- und HPLC-Verfahren) zur Bestimmung der Enantiomerenverhaeltnisse steht als Alternativmethode **die** duennschicht-chromatographische Racemattrennung auf der Basis des Ligandenaustauschs mit einer DC-Fertigplatte zur Verfuegung. Hierbei bilden **die** Antipoden mit einem **Metallion** (z.B. Cu(++)) und dem chiralen Absorbens diastereomere Assoziate, deren unterschiedliche Stabilitaet **die** Trennung ermoeeglicht. Optimale Resultate mit einer DC-Fertigplatte wurden erzielt bei aufsteigender, eindimensionaler Entwicklung in einer Trogkammer mit Kammersaettigung. **Die** mobile Phase war ein ternaeres Gemisch aus **Methanol**, Wasser und Acetonitril. Aufgetragen wurden 2 Mikroliter einer 1 prozentigen Loesung des Racemates. **Die** Laufstrecke betrug 13 cm, **die** Laufzeit 30 bzw. 90 Minuten. Der Nachweis erfolgte mit 0,1 %iger Ninhydrin-Reagens. Mit einigen Trennbeispielen wird **die** Trennmethode bestaetigt. (Haas)

29/3,AB/42 (Item 9 from file: 315)
 DIALOG(R)File 315:ChemEng & Biotec Abs
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182588 CEABA Accession No.: 17-12-016646 DOCUMENT TYPE: Journal
 Title: The structure of the CuZnO catalyst by an in-situ EXAFS study
 AUTHOR: Tohji, K.; Udagawa, Y.; Mizushima, T.; Ueno, A.
 JOURNAL: Journal of Physical Chemistry, Volume: 89, Issue: 26, Page(s): 5671-5676
 CODEN: JPCHAX ISSN: 0022-365
 PUBLICATION DATE: 1985 (850000) LANGUAGE: English
 ABSTRACT: Bei der fuer **die Methanolsynthese** industriell wichtigen CuZnO-Katalysatoren (mit Al₂O₃ oder Cr₂O₃) ist **die** Struktur des aktiven Kupfers noch umstritten. Der Katalysator wird bei ca. 550 K eingesetzt und verliert bei hoher Temp. an Aktivitaet. **Die** Untersuchung zeigt, dass sich **die** Struktur im H₂-Strom bei Erwaermungs-Abkuehlungszyklen bis 550 K reversibel aendert. Oberhalb 400 K liegen disperse **Cu-Metallcluster** vor, d.h. diese sind katalytisch aktiv, unter 400 K **Cu-Metallcluster** mit Sauerstoffbindung. Bei hoeherem Erhitzen koagulieren **die** Cluster irreversibel zu grossen kristallinen Teilchen. Demnach ist es wichtig, dass Katalysatorcharakterisierungen unter den Anwendungsbedingungen durchgefuehrt werden. (Weckend)

29/3,AB/43 (Item 10 from file: 315)
 DIALOG(R)File 315:ChemEng & Biotec Abs
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177291 CEABA Accession No.: 17-12-011349 DOCUMENT TYPE: Journal

Title: **Methanol** synthesis reaction over **copper**-group IV
metal amorphous alloys as catalyst precursor

AUTHOR: Shibata, M.; Ohbayashi, Y.; Kawata, n.; Masumoto, T.; Aoki, K.

JOURNAL: Journal of Catalysis, Volume: 96, Issue: 1, Page(s): 296-298

CODEN: JCTLA5 ISSN: 0021-951

PUBLICATION DATE: 1985 (850000) LANGUAGE: English

ABSTRACT: **Die Methanolsynthesereaktion** ueber amorphen Kupfer-Gruppe IV-Metall-Legierungen als Katalysator-Precursor und der Aktivierungsprozess der amorphen Legierung werden beschrieben. Kupfer (99,9 %) und eine Gruppe IV-Metall (99,9 % Ti, 99,6 % Zr, 96 % Hf) werden in Argonatmosphaere zusammengeschmolzen. Aus einer amorphen Cu70Zr30-Legierung werden CuZr-Kristalle durch 30-minuetiges Erhitzen auf 873 k im Vakuum erhalten. **Die** kristalline Phase wird durch Roentgenbeugung identifiziert. Eine aktive Position der amorphen Cu-Zr-Legierung stellt Cu(+) dar (vergleichbar zum CuZnO-Katalysator). (Schoeffmann)

29/3,AB/44 (Item 11 from file: 315)

DIALOG(R)File 315:ChemEng & Biotec Abs

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160252 CEABA Accession No.: 16-12-010723 DOCUMENT TYPE: Journal

Title: Studies on the chemical state of Cu during **methanol** synthesis

AUTHOR: Fleisch, T.H.; Mieville, R.L.

JOURNAL: Journal of Catalysis, Volume: 90, Issue: 1, Page(s): 165-172

CODEN: JCTLA5 ISSN: 0021-951

PUBLICATION DATE: 1984 (840000) LANGUAGE: English

ABSTRACT: Ein kommerzieller CuOZnAl2O3-Katalysator wird 1 Stude bei 250 Cel und 2 atm einem Gas folgender Zusammensetzung ausgesetzt: 73 % H2, 25 % CO, 2 % CO2. Es findet eine quantitative Umwandlung aller Cu(2+)-Ionen in **Cu-Metall** statt. Es liegt nur eine Menge kleiner als 2 % des gesamten Cu als Cu(+)-Ion vor. Cu(+) kann aus diesem Grunde nicht **die** aktive Cu-Position des CuOZnAl2O3-Katalysators sein. XPS-Daten werden bezueglich der Katalysatoroberflaeche angegeben. (Schoeffmann)

29/3,AB/45 (Item 12 from file: 315)

DIALOG(R)File 315:ChemEng & Biotec Abs

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095322 CEABA Accession No.: 12-12-009055 DOCUMENT TYPE: Journal

Title: Comparative **study** of **copper** and nickel surfaces for the decomposition of **methanol** using ultraviolet photoelectron spectroscopy

AUTHOR: Kojima, I.; Sugihara, H.; Miyazaki, E.; Yasumori, I.

JOURNAL: Journal of the Chemical Society, Faraday Transactions 1, Volume: 77, Issue: 6, Page(s): 1315-1329

CODEN: JCFTAR ISSN: 0300-959

PUBLICATION DATE: 1981 (810000) LANGUAGE: English

ABSTRACT: Im UV-Photoelektronen-Spektroskopieverfahren verglich man **die** katalytischen Eigenschaften von Kupfer- und Nickeloberflaechen. Untersucht wurden **die** Adsorption, sowie der Zerfall von Kohlenmonoxid, **Methanol**, Formaldehyd und Methylformat im Temperaturbereich von 78 bis 400 K. Bei der molekularen Adsorption von **Methanol** und Formaldehyd an Kupferoberflaechen bei 295 Grad K und an Nickeloberflaechen bei 170 Grad K waren chemische Oberflaechenwechselwirkungen ueber Sauerstoff-Einzelpaar-Orbitale festzustellen. Durch nachfolgendes Einwirken von **Methanol** und Methylformat auf Kupferoberflaechen bei 295 Grad K wurde **die**

Bildung von adsorbiertem Formaldehyd bewirkt. An einer Nickel-Oberflaeche jedoch erfolgte bei 78 Grad K die schrittweise Abwanderung von Wasserstoffatomen zu CO und H₂. (Autor)

29/3,AB/46 (Item 1 from file: 103)
 DIALOG(R)File 103:Energy SciTec
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03730762 DE-94-0GG353; EDB-94-146728

Title: Catalytic combustion of solvent containing air on base metal catalysts

Original Title: Katalytische Nachverbrennung loesemittelbeladener Abluft an Nichtedelmetallkatalysatoren

Author(s)/Editor(s): Lintz, H.G. (Karlsruhe Univ. (T.H.) (Germany). Inst. fuer Chemische Verfahrenstechnik); Wittstock, K. (Karlsruhe Univ. (T.H.) (Germany). Inst. fuer Chemische Verfahrenstechnik)

Corporate Source: Kernforschungszentrum Karlsruhe GmbH (Germany). Projekt Europaeisches Forschungszentrum fuer Massnahmen zur Luftreinhaltung (PEF)

Sponsoring Organization: Land Baden-Wuerttemberg, Stuttgart (Germany); Commission of the European Communities, Brussels (Belgium)

ISSN: 0931-2749

Publication Date: Jun 1994

(73 p)

Report Number(s): KFK-PEF-121

Order Number: DE95702865

Contract Number (Non-DOE): Project Number PEF 3 91 003

Language: German

Abstract: The catalytic combustion is one of several industrial processes for pollution abatement. The aim is to convert volatile organic compounds (VOC) to carbon dioxide and water. Usual catalysts are noble metals like platinum and palladium or oxides of base metals such as copper manganate and **copper** chromite. The **study** of the catalytic combustion of solvent containing air has shown, that not only the desired total oxidation to carbon dioxide and water, but also the formation of partially oxidized intermediates takes place. These by-products are often more noxious than the original solvent itself. With the knowledge of the reaction network and the reaction rates it is possible to design a reactor in a way, that the concentrations of each compound, either solvent or intermediate, are less than the limit. This present study shows experimental verification for the case of the oxidation of the solvents butyl acetate, **isopropyl alcohol** and acetone on copper managanate catalysts. (orig.). 27 figs., 8 tabs., 30 refs.

Non-English Abstract: **Die** katalytische Nachverbrennung (KNV) ist eines von mehreren in der industriellen Praxis angewendeten Verfahren zur Abluftreinigung. Dabei sollen fluechtige organische Komponenten (VOC) zu Kohlendioxid und Wasser verbrannt werden. Als Katalysatoren werden Edelmetalle wie Platin und Palladium, oder aber Oxide von Nichtedelmetallen, z.B. Kupfermanganat und -chromit, verwendet. **Die** Untersuchung der katalytischen Nachverbrennung loesemittelbeladener Abluft an Nichtedelmetallkatalysatoren hat gezeigt, dass nicht nur **die** erwuenschte Totaloxidation zu Kohlendioxid und Wasser ablaeuft, sondern auch partiell oxidierte Produkte gebildet werden koennen, **die** aufgrund ihrer Toxizitaet oder Geruchsintensitaet ebenfalls Grenzwerten unterliegen. Bei Kenntnis des Reaktionsnetzwerks und der Reaktionsgeschwindigkeiten kann ein Nachverbrennungsreaktor so dimensioniert werden, dass ein nahezu vollstaendiger Ausbrand aller Schadstoffkomponenten, einschliesslich der neu gebildeten Zwischenprodukte, sichergestellt ist. **Dies**

konnte im Rahmen dieser Arbeit beispielhaft fuer **die** Oxidation der Loesemittel Butylacetat, i-Propanol und Aceton an Kupfermanganat-Katalysatoren gezeigt werden. (orig.)
Abstract Language: Deutsch

29/3,AB/47 (Item 2 from file: 103)
DIALOG(R)File 103:Energy SciTec
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02775422 DE-89-013134; EDB-89-166466
Title: Method for the production of **methanol** and higher alcohols, and description of a suitable catalyst system
Original Title: Zink-Chrom-Kupfer-Katalysator und dessen Verwendung fuer **die** Alkoholsynthese
Author(s)/Editor(s): Fattore, V.; Notari, B.; Paggini, A.; Lagana, V.
Patent No.: DE 3244313 C2
Patent Assignee(s): Snamprogetti SpA, Milan (Italy)
Priority No.: IT 25390A-81
Patent Priority Date: 2 Dec 1981
Publication Date: 6 Feb 1986
(6 p)
Language: In German
Abstract: Catalyst system and its use for the production of **methanol** and higher alcohols from synthesis gas, the catalyst system containing the elements zinc, chromium, **copper**, alkaline **metal**(s) and, if necessary, molybdenum, manganese, lanthanum, cerium, aluminium, titanium and/or vanadium and these elements being wholly or partly chemically bonded to oxygen and/or to each other. (orig.).
Non-English Abstract: Katalysatorsystem und dessen Verwendung zur Herstellung von **Methanol** und hoeheren Alkoholen aus Synthesegas, wobei das Katalysatorsystem **die** Elemente Zink, Chrom, Kupfer, Alkalimetall(e) und gegebenenfalls Molybdaen, Mangan, Lanthan, Cer, Aluminium, Titan und/oder Vanadium enthaelt und diese Elemente ganz oder teilweise chemisch an Sauerstoff und/oder untereinander gebunden sind. (orig.). Abstract Language: Deutsch

29/3,AB/48 (Item 3 from file: 103)
DIALOG(R)File 103:Energy SciTec
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01383803 NET-84-90177; EDB-84-081600
Title: The determination of acidic degradation products in aqueous ethylene **glycol** and propylene **glycol** solutions using ion chromatography
Author(s): Rossiter, W.J.; Brown, P.W.; Godette, M.
Affiliation: National Bureau of Standards, Washington, DC (USA)
Source: Sol. Energy Mater. (Netherlands) v 9:3. Coden: SOEMD
Publication Date: Oct 1983
p 267-279
Language: English
Abstract: Aqueous ethylene **glycol** and propylene **glycol** solutions are the principal antifreezes used as heat transfer fluids in flat-plate solar collector systems. Acidic degradation products formed in the thermal oxidation of these solutions have been analyzed using ion chromatography (IC) and the ion-chromatography exclusion (ICE) method of analysis. The primary acidic degradation product found in the case of ethylene **glycol** solutions was glycolic acid. Propylene **glycol** solutions yielded mainly lactic acid. The presence of **metallic copper** significantly increased the

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amount of degradation products over that produced in the absence of metal. Solutions heated in the presence of metallic aluminium were only slightly more degraded than those heated without metal, but contained relatively large amounts of formic acid. Only traces of formic acid were observed when the **glycol** solutions were heated in the absence of metal or in the presence of **metallic copper**.
(A.V.)